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ORGANIC CHEMISTRY:
A BRIEF COURSE

by ROBERT WARD GETCHELL
Professor of Chemistry Emeritus, Iowa State Teachers College

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*Gilmont, R. *Anal. Chem.* **20**, 1109 (1948)

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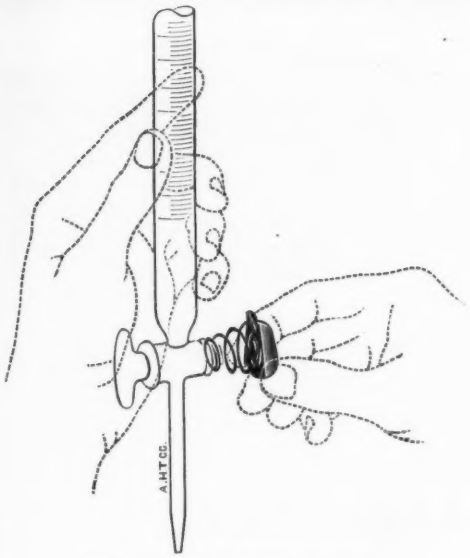

3291-B. Method of attachment or removal.

• **STOPCOCK TENSION CLIPS, A.H.T. Co. Specification (Patent Applied For).** Of Stainless steel, for pressure-tight seating of practically any type stopcock with groove for rubber washer at small end of plug. Can be attached instantly by compression of the spring and with thumb and finger pressure on the narrow flanged sides. Cannot work loose, but can be removed instantly. Recommended for glassware which requires hand or mechanical shaking such as Van Slyke Apparatus, Separatory Funnels, etc., and to improve the efficiency of stopcocks on Burettes, Gas Analysis Apparatus, Sampling Tubes, etc.

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3291-B. In position on stopcock plug of a Burette.

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3291-B.
Rear view of Stopcock Tension Clip showing spring opposed jaws.

3291-B.
Side view of Stopcock Tension Clip.

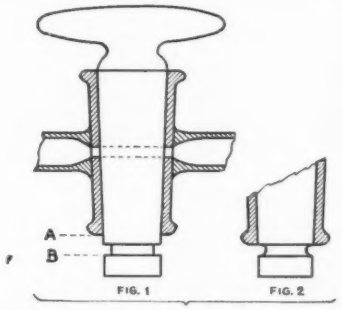

Fig. 1. Showing correct type of stopcock for satisfactory use with Clip, i.e., with right-angled groove and with distance A–B of ⅛-inch or more.

Fig. 2. Showing unsatisfactory type of stopcock for use with Clip, i.e., with groove too close to shell and with shallow, rounded groove which tends to eject the spring.

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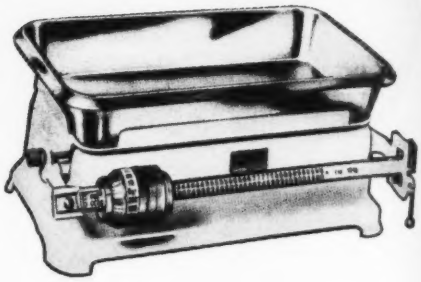

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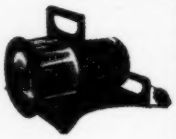

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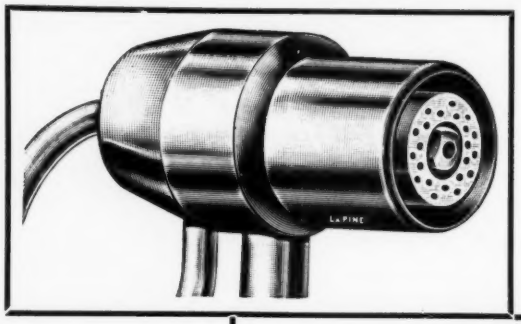

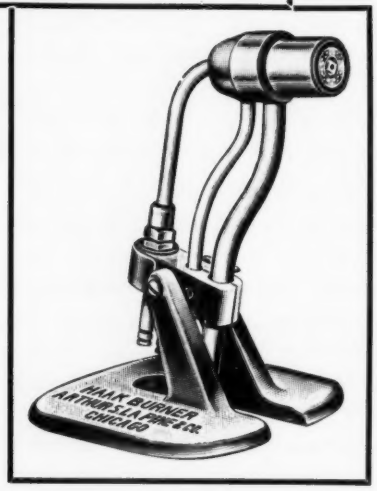

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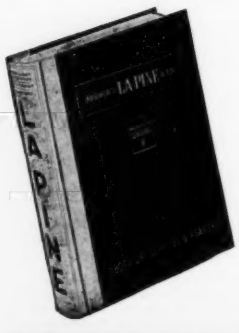

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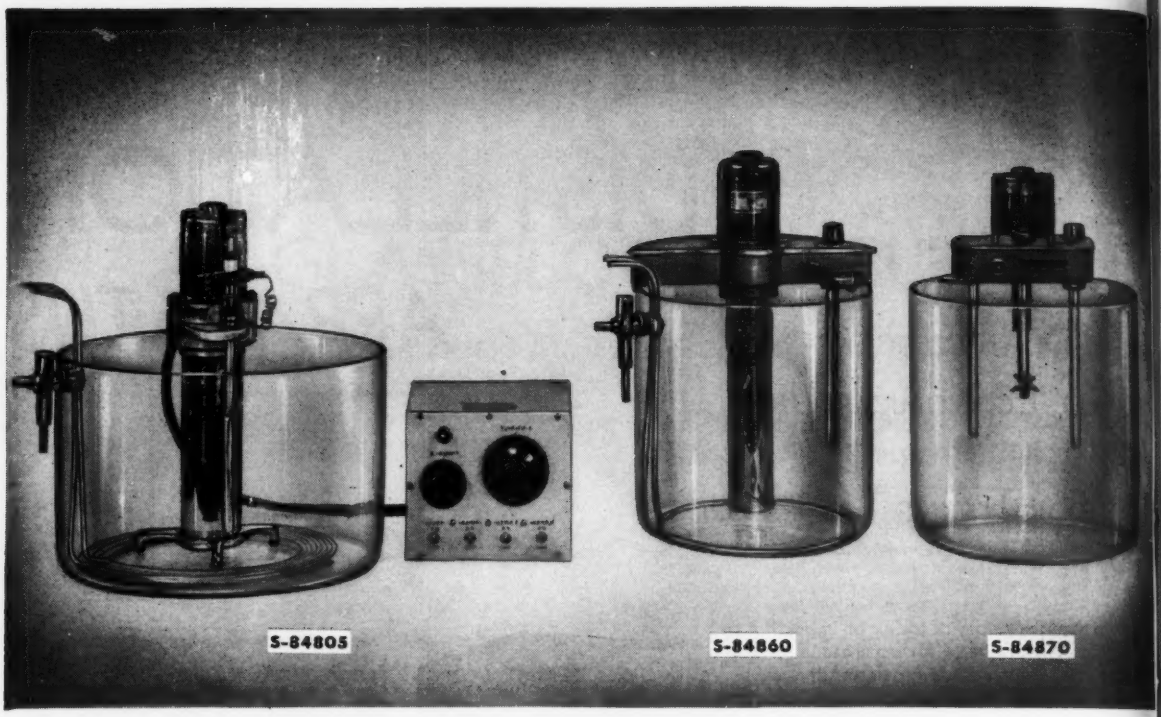

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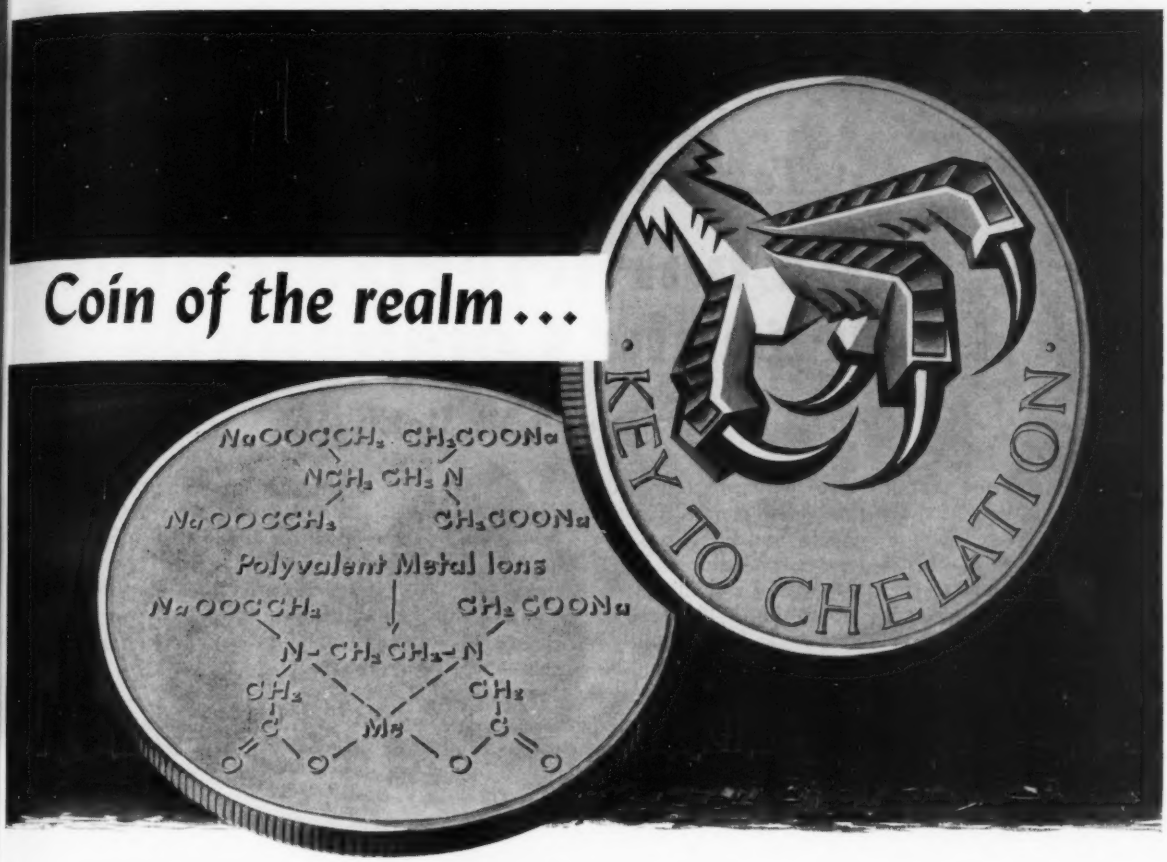

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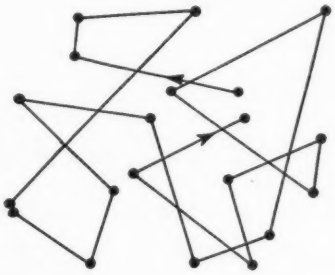

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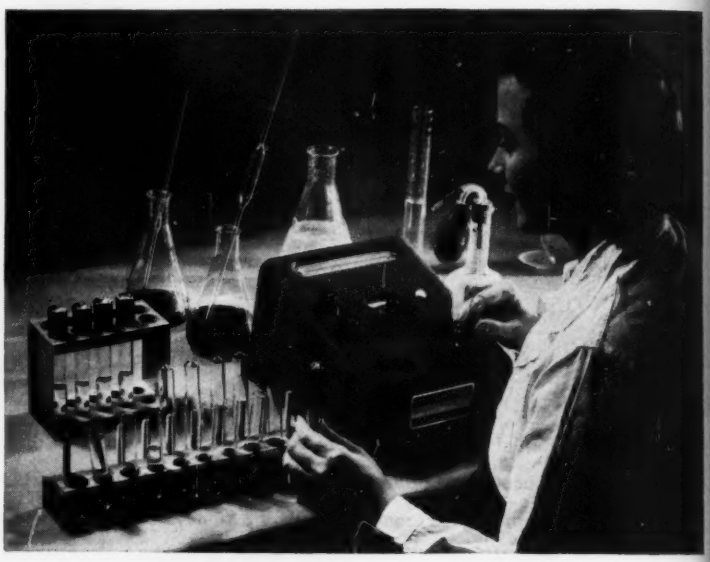

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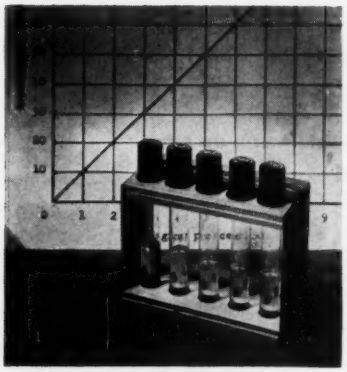

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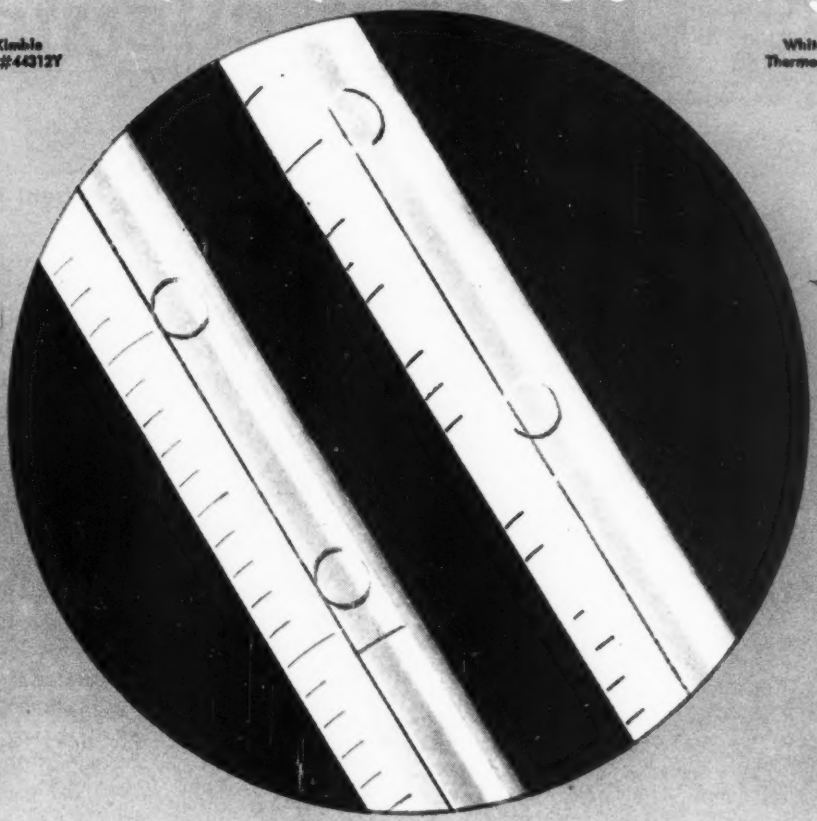

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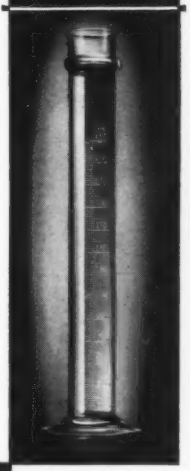

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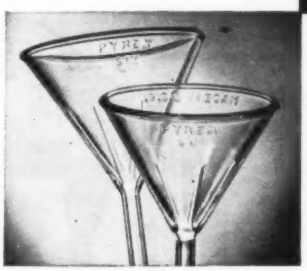

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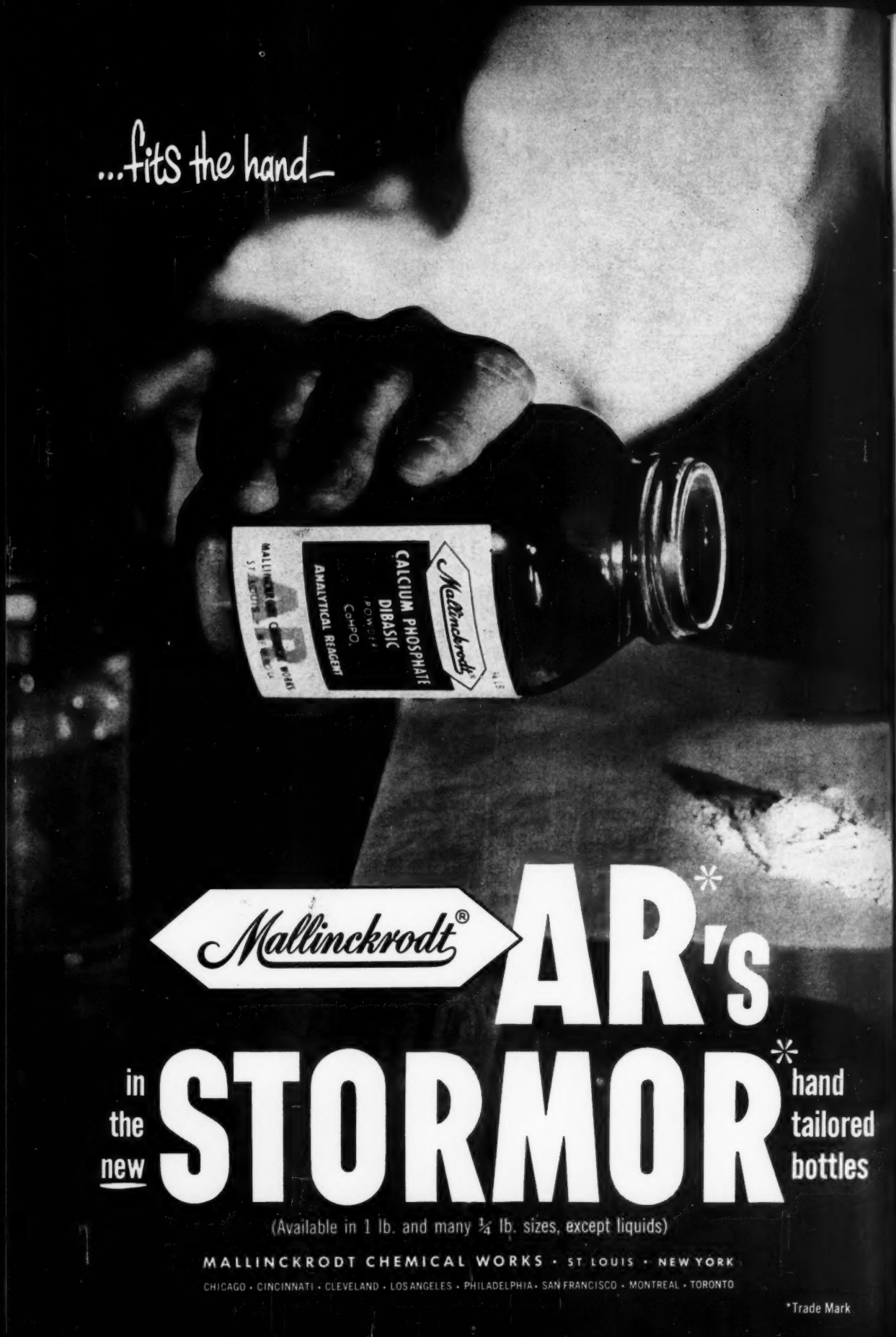
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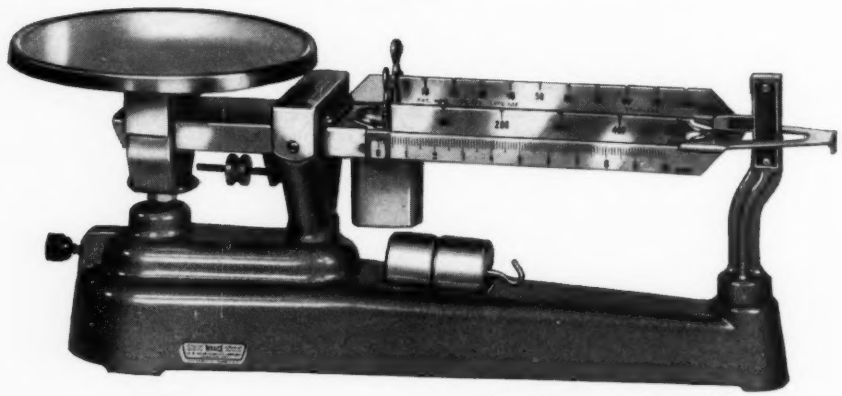

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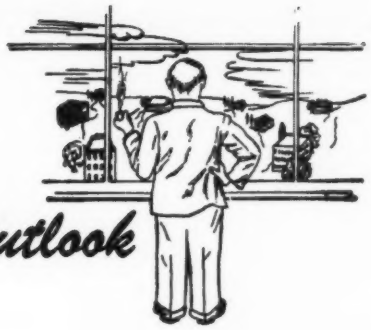


Editor's Outlook

WAY back in the days when the now-well-known objective tests were new, a speaker was discussing their virtues. A disgusted listener was heard to remark: "I would just as soon grade my students in the order in which they left the room as by any such tests as these." The speaker's response was that he would, too, if there was reason to believe that the students left the room in the order of their accomplishment.

Which of course brings out the point that the validity of any scale of measurement depends upon the relation between the observed graduations of the scale and the variation in the property being measured. Unless he knew something of the details, who would believe that he could tell the composition of a metal alloy by observing the position of the hands of a clock?

Not long ago a faculty group was discussing the evaluation of good teaching and the qualities of a good teacher. Who can tell, with any degree of objectivity, whether a teacher is doing a good job? Some put faith in questionnaires of student opinion; some believe that a department head will undoubtedly be able to judge his colleagues. Some believe that teaching ability is closely correlated to research accomplishment; some think that good teachers are born that way and there is nothing further to do about it, anyway.

But I was especially intrigued by one suggestion: A good teacher is generally distinguished by his ability to tell a good story! After all, what are the marks of a good story teller? An appreciation of relative values, particularly of human relations; an instinct for the dramatic; an ability to reduce a situation to understandable terms; a desire to share his experiences and enthusiasms with others; and above all a sense of humor. These, too, are among the important attributes of a good teacher. I believe that if we think in retrospect we will realize that the teachers we remember most fondly will rank high on this empirical scale.

But, of course, not all. As someone has said: "No generalization—not even this one—is universally true." The same teacher will not be equally effective on two different minds, nor indeed upon the same mind at different stages in its development. Teachers who may be wonderfully successful in elementary classes sometimes fail miserably in more advanced ones, and vice versa. We are too prone to a theatrical concept of teaching—a big lecture hall with good acoustics, where crowds of students are pleasantly entertained, and stimulated to at least a modicum of intellectual response. This takes skill on the part of the teacher, but it is only one kind of good teaching. It is conceivable that the good teacher may be almost inarticulate and produce his result by gentle suggestion or by example. He may even shrink from the kind of warm human contact which makes his more popular colleague effective. By doing his work with single individuals, or small groups, he may leave a more lasting and more fundamental impression. He, too, can be a good teacher.

So the matter is not so simple. But, after all, do we not always approach truth by a series of approximations?

ASPECTS OF ISOMERISM AND MESOMERISM

III. Stereoisomerism

RICHARD L. BENT
Eastman Kodak Company, Rochester, New York

THE field of stereoisomerism is so extensive that a review article of reasonable length has to be quite limited in scope. It is the object of the present paper to give an elementary but up-to-date account of the basic concepts of stereoisomerism. Nevertheless, no attempt has been made to discuss as many different types of isomerism as possible. Only those topics which are of particular interest to the organic chemist are included, and only the stereochemistry of the element carbon is considered to any extent. The distinction between molecular and statistical optical activity is emphasized throughout, because of the inadequate treatment given the subject in the majority of texts.

PRELIMINARY REMARKS

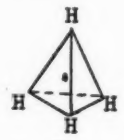
In regard to the carbon compounds, the basic problem is, of course, the determination of the spatial arrangements of the atoms, or groups, about each carbon atom. Neglecting atomic vibrations, it is found that in some molecules, *e. g.*, methane and cyclopropane, the atoms occupy fixed positions in space (relative to each other). The majority of compounds, however, do not consist of molecules possessing fixed configurations because of the presence of various groups of atoms which are free (or relatively free) to rotate about a single bond, *e. g.*, ethane, toluene, etc. Although the spatial distribution of atoms is usually three-dimensional, as in methane, ethane, etc., it may be planar, as in ethylene and benzene, or even linear, as in acetylene. Stereochemical theory can, in general, show what the particular distribution is, although it cannot tell us the exact dimensions involved, without the help of the physicist. As described in Part I, the spatial arrangements of the atoms in a molecule are portrayed on paper by means of configurational formulas.

When two or more molecules possess identical molecular formulas, they are said to exhibit isomerism. While structural isomerism is concerned with molecules which possess the same molecular formula but different structural formulas (see Part II), stereoisomerism (and this includes both optical and geometric isomerism) is concerned with molecules possessing identical structural formulas but different configurational formulas.[1] Molecules exhibiting structural isomerism differ in regard to which atom is linked to which; molecules exhibiting stereoisomerism, on the other hand, do not differ in this respect. The differences between stereoisomeric molecules can only be explained by considering the spatial relationships of the atoms.

TETRAHEDRAL NATURE OF CARBON

Although Pasteur and Wislicenus made considerable contributions to stereochemical theory, Le Bel and Van't Hoff in 1874 (independently of each other) were the first to treat the subject in a thorough and systematic manner. Essentially, both their theories were the same, although Le Bel elaborated on Pasteur's conception of molecular asymmetry, while Van't Hoff extended Kekulé's theory of the quadrivalency of carbon and was more concerned with the strict geometrical aspects of molecules. In fact, Van't Hoff's theory was essentially a spatial interpretation of Kekulé's theory.[2]

Without considering historical details, the discussion that follows can be regarded as the present attitude toward the basic concepts of stereochemistry. For simplicity, let us consider the methane molecule. The carbon atom in such a molecule is depicted as being at the center of a regular tetrahedron (a solid figure of four sides, each of which is an equilateral triangle), while the hydrogen atoms are considered as being located at the four corners of the tetrahedron, as shown in I (the central carbon atom, portrayed here by a dot, is often omitted in such diagrams). Although a tetrahedral arrangement was not the only arrangement capable of accounting for the apparent equality of the four carbon valencies, it was originally adopted on the basis of chemical evidence.



(I)

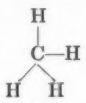
The hydrogen atoms in methane are linked by covalent bonds to the central carbon atom, each bond con-

[1] The detailed relationships that exist between the different types of stereoisomerism (as well as structural isomerism) are shown in the chart presented in Part II of this series (June issue, p. 284). It should be consulted throughout this paper.

[2] For a discussion of the two theories, see FINDLAY, A., "A Hundred Years of Chemistry," 2nd ed., Gerald Duckworth and Co., Ltd., London, 1948, pp. 55–68.

sisting of a shared pair of electrons. Because of the extremely high velocity of the electrons, each hydrogen atom can be considered to be actually *joined* to the carbon by a cloud of negative electricity (or electron cloud) extending around the respective nuclei and, in particular, between the nuclei. Inherent directional properties are associated with a covalent bond (in contrast to an electrovalent bond). Each covalent bond can be considered as extending directly from one atom to another and, in general, when two or more such bonds are attached to a single atom, the bonds are at definite angles to each other.[3]

A covalent bond can be simply represented on paper by means of a straight line. In diagram II we have an alternative picture of the methane molecule, one in which all of the bonds and atoms are represented. Actual molecular models, the so-called ball-and-peg type, can readily be constructed from the valence-bond type of formula II, and have been widely used for



(II)

demonstration purposes (see Figure 1). The interatomic distances are usually exaggerated in this type of molecular model, however, and often no allowance is made for the different sizes of atoms. These defects are not present in the Fisher-Hirschfelder-Taylor (or Stuart) molecular models (see Figure 2).

Since a considerable part of stereoisomerism is concerned with the *relative* spatial arrangements of the atoms in a molecule, rather than with the exact sizes of the atoms and the exact interatomic distances, the ball-and-peg type can often give us the information desired. However, in order to understand the stereoisomerism that occurs in certain derivatives of biphenyl (or any other type of stereoisomerism that depends upon steric hindrance), a knowledge of atomic sizes and bond lengths is important. For problems of this kind, Fisher-Hirschfelder-Taylor models should be used.

Because the carbon atom in methane is at the center of a regular tetrahedron, the angle subtended by any two carbon-hydrogen bonds, the so-called tetrahedral angle, is easily calculated to be approximately 109° 28′.[4]

By means of X-ray and electron-diffraction experiments, it has been shown that the tetrahedral angle does not vary appreciably from this value when all four atoms, or groups, are similar in nature. When they are not similar, however, distortions of a few degrees can occur, because of differences in the repulsive and attractive forces of the particular groups. Thus, the Cl—C—Cl bond angle in methylene chloride is found to be about 112°.[5]

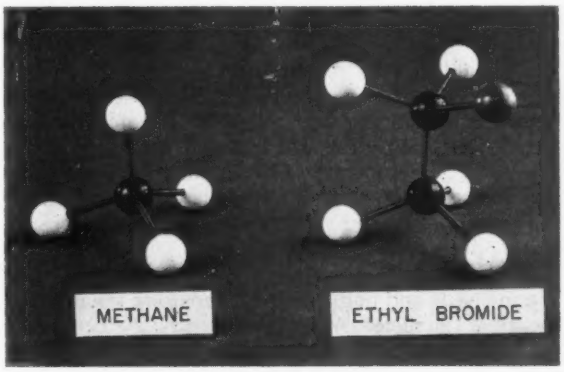


Figure 1

In the cyclic compounds possessing very small rings, the constancy of the tetrahedral angle is not even approximately preserved. The angle is forced to assume smaller values for purely "geometric" reasons. Thus, in cyclopropane and cyclobutane, both of which possess a planar arrangement of carbon atoms, the carbon-carbon bond angles are 60° and 90° respectively. It was originally assumed that the next higher homologue, cyclopentane, also possessed a planar ring, and that the carbon-carbon bond angle was approximately 108°, deviating only slightly from the tetrahedral angle.

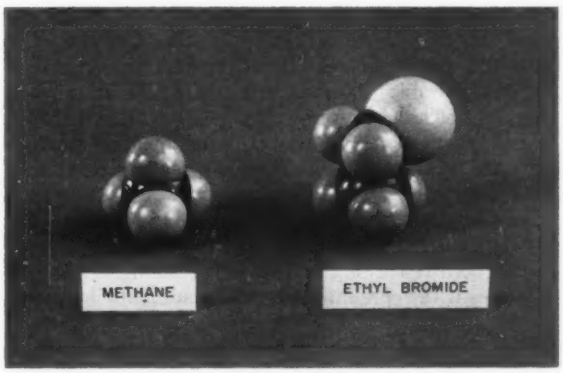


Figure 2

However, it is now believed that the cyclopentane ring is slightly puckered.[6] In the case of cyclohexane, it appears that the tetrahedral angle is not distorted at

[3] Carbon has four electrons in its outer (valence) shell, two *2s* electrons and two *2p* electrons. How these four electrons (possessing different energies and spatial distributions) are able to give rise to four equivalent bonds (as in methane) is explained by quantum mechanics. For a nonmathematical explanation, and for a general treatment of stereochemistry in terms of molecular-orbital theory, see MILLS, W. H., *J. Chem. Soc.*, **1942**, 457; COULSON, C. A., *Quart. Revs.*, **1**, 144 (1947); NOLLER, C. R., J. CHEM. EDUC., **27**, 504 (1950).

[4] For a delightfully simple calculation, see BRITTIN, W. E., J. CHEM. EDUC., **22**, 145 (1945).

[5] See WHELAND, G. H., "Advanced Organic Chemistry," 2nd ed., John Wiley & Sons, Inc., New York, **1949**, p. 167.

[6] KILPATRICK, J. E., K. S. PITZER, AND K. SPITZER, *J. Am. Chem. Soc.*, **69**, 2483 (1947).

all in the so-called "chair" form, but that slight distortions probably occur in the "boat" form; considerable distortion occurs, of course, in the intermediate planar form (all forms being in dynamic equilibrium with one another).[7] There is little doubt that the tetrahedral angles in cycloheptane and cyclooctane are distorted to a certain extent; similarly, though to a lesser extent, in the higher homologues, until rings of considerable size (16 carbon atoms or so) have been reached.

It should be pointed out that there are two major sources of "internal strain" in the cycloalkanes:[8] (*a*) distortions of the (ring) bond angles from the normal tetrahedral value (in regard to small rings this corresponds to Baeyer's classical strain theory); (*b*) deviations of the carbon-hydrogen spatial arrangements from the preferred staggered positions (preferred because of the decrease in mutual repulsion of the atoms). Both types of strain may be present at the same time in a molecule and both result in increasing the internal energy of the molecule.

In the three- and four-membered rings, the considerable distortion of the bond angles appears to be the major source of the strain present. The second factor, the so-called "constellation" factor, is small because the small angles increase the distances between the hydrogen atoms and therefore decrease repulsion. In a five-membered ring, the constellation factor is of major importance. The mutual repulsions of the atoms in a strictly planar cyclopentane would result in a large increase in internal energy. However, the strain (*i. e.*, the "constellation" strain) is decreased to a certain extent by rotations of the carbon atoms, causing distortions of the bond angles, and resulting in a nonplanar molecule. While cyclohexane (particularly in the chair form) can exist in a relatively strain-free configuration, with all carbon-hydrogen bonds nicely staggered, this is not possible for cycloheptane or cyclooctane, and as a result the latter compounds possess considerable internal strain (molecular models show appreciable overcrowding of the atoms). The strain appears to decline gradually for the higher members until a constant value is reached at approximately cyclohexadecane.

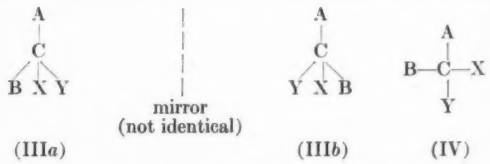
Because almost every statement that is made concerning the spatial aspects of a molecule is complicated by the occurrence of freedom of rotation within the molecule, a few preliminary remarks should be made at this point.

Thus, it is often stated that a molecule like ethane, for example, does not possess any isomers. More exactly, this means no separable isomers. Because of the rotation (or rotation-oscillation) of the methyl groups, there are, of course, an infinite number of different spatial positions possible for the two groups, each "instantaneous" form theoretically being an isomer and displaying different properties (actually, because ethane is a relatively symmetrical molecule, one should only consider the different spatial positions during one third of a rotation since the configurations repeat themselves during a complete rotation). The properties exhibited by an appreciable number of molecules will, of course, be the average of the properties of each individual molecule. Because one is normally concerned with an immense number of molecules this average does not vary to an appreciable extent (as shown by probability theory). In effect, therefore, we are dealing with one substance only.

Again, when the statement is made that a compound can exist in only (say) two stereoisomeric forms, this usually means two "average" forms. If freedom of rotation is present, both of these isomers (considered individually) actually consist of a vast number of different spatial forms. The observed properties of each isomer will, therefore, be the result of the averaging of the properties of each of these individual structures. Thus, in effect, we are dealing with only two different substances.

If no freedom of rotation is present in a molecule, it is obvious that only one spatial formula is capable of representing it, *e. g.*, in III*a*, if A, B, X, and Y are all single atoms. If, however, these letters represent groups of atoms, capable of rotation, then there are an infinite number of spatial formulas capable of representing the substance. In practice, of course, one of these "static" formulas is chosen to represent the molecule, the freedom of rotation being taken for granted (see Part I).

The chemical evidence for a tetrahedral environment should now be examined. Let us consider a fully substituted methane. Isomerism only occurs when all four of the atoms or groups about the central carbon are different. A carbon atom connected in this manner is said to be *asymmetric*. Only two isomers, III*a* and III*b*, are possible for a molecule of the type CABXY,



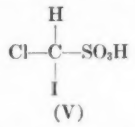
(III*a*) (III*b*) (IV)

where A, B, X, Y (here, and throughout this paper) represent dissimilar atoms or "simple" groups, *i. e.*, the attached groups must not contain further assymmetric centers or be capable of exhibiting any other type of isomerism, otherwise more than two isomers will be possible. Since both molecules possess the same structural formula, IV, or CABXY for short, but have different spatial, or configurational formulas, III*a* and

[7] Recent physical evidence (infrared spectra, Raman spectra, etc.) indicates that at normal temperature cyclohexane is substantially in the more symmetrical chair form, together with a small percentage of the less symmetrical boat form (the latter possessing a higher energy content). The number of molecules possessing the boat form, however, increases rapidly with the temperature. See BECKETT, C. W., K. S. PITZER, and R. SPITZER, *ibid.*, **69**, 2488 (1947).

[8] The above discussion is largely based on an article by BROWN, H. C., AND M. BORKOWSKI, *ibid.*, **74**, 1894 (1952), and on certain of the references given there. See also discussion by WHELAND, G. H., ref. 5, p. 365.

III*b*, they are called stereoisomers of one another. Because they exhibit a nonsuperimposable mirror-image relationship to each other,[9] and because an appreciable number of molecules of each isomer rotates the plane of polarized light to an equal but opposite degree, they are specifically called *enantiomorphs* (from the Greek words *enantio*, meaning opposite, and *morphos*, meaning shape or form). Chloroiodomethanesulfonic acid, V, is one of the simplest examples of this type of isomerism. As expected from the tetra-



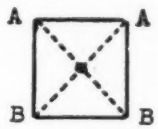
(V)

hedral theory, this compound exists in two enantiomorphic forms. Other well-known examples are: lactic acid, $CH_3C^*HOHCOOH$; malic acid, $HOOCC^*HOHCH_2COOH$; and "active" amyl alcohol, $C_2H_5C^*H(CH_3)CH_2OH$. Each of these compounds exists in two, and only two, enantiomorphic forms because of the presence of a single asymmetric carbon atom (denoted by an asterisk).

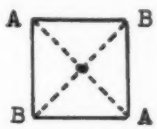
The terms *asymmetry* and *asymmetric* are used throughout this paper because of their familiarity, rather than the more correct terms *dissymmetry* and *dissymmetric*. In common usage, an asymmetric molecule is considered to be one that cannot (at any time) be superimposed upon its mirror image, *i. e.*, it is a molecule that is capable of possessing an enantiomorphic partner. Since any molecule containing a single carbon atom linked to four different atoms or groups can exist in two enantiomorphic forms,[10] we speak also of an asymmetric carbon atom. Thus, the lactic acid molecule is asymmetric because it possesses a nonsuperimposable mirror image; one of the carbon atoms is attached to four different groups (CH_3, H, OH, and COOH) and therefore the molecule also possesses an asymmetric atom.

Strictly speaking, however, the word asymmetry implies the absence of *all* elements of symmetry (see later). If a molecule is to be capable of having an enantiomorphic partner, it has been found that there are only *three* elements of symmetry that must at all times be absent (the plane, point, and alternating axis of symmetry). It is still possible, however, for certain enantiomorphic molecules to possess what is termed an *ordinary* axis of symmetry, even though the all-important elements are absent, *i. e.*, an optically active molecule need not be devoid of all elements of symmetry. Thus, a molecule is more correctly said to be dissymmetric if it is not superimposable on its mirror image, and asymmetric if it does not possess *any* of the elements of symmetry (including the ordinary axis). It is correct, however, to speak of a single carbon atom attached to four different groups as being asymmetric since it cannot possess any of the elements of symmetry.[11]

Let us suppose that the atoms or groups in a substituted methane were distributed in a planar rather than a tetrahedral manner. The number of isomers predicted in such cases is at complete variance with the number of isomers that actually exists.[12] Thus, in a molecule of the type CAABB, for example, two isomers, VI*a* and VI*b*, would be predicted (the dot at the



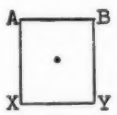
(VI*a*)



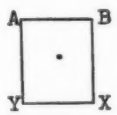
(VI*b*)

center represents a carbon atom and the radiating dotted lines the valence bonds). No such isomers have ever been discovered with carbon as the central atom. It is of interest to note, however, that planar structures of this type occur in certain compounds of platinum, for example, when the latter is quadrivalent.

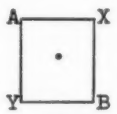
When we come to a substituted methane of the type CABXY, where all four of the substituted atoms or groups are different, three planar isomers, VII, VIII, and IX, would be expected rather than the two tetrahedral isomers, III*a* and III*b*, that actually exist. It



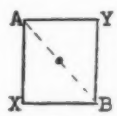
(VII)



(VIII)



(IX)



(X)

may appear to the reader that there are other arrangements of the atoms or groups which would give rise to additional planar isomers, but with all possible arrangements it is found that by moving the molecule in various ways a structure identical with VII, VIII, or IX always results. Thus, IX and X might seem to be two distinct structures, but on rotating X about the AB axis, Structure IX results.

To summarize: For a *tetrahedral* carbon atom connected to four different groups, one predicts and finds two stereoisomers only, and specifically two *enantiomorphic* isomers. For a *planar* carbon atom connected

[9] If A, B, X, and Y represent atoms, this relationship is obvious. Nevertheless, even if they represent groups, it is still true that for every possible position (during rotation of the groups) the corresponding mirror-images will be nonsuperimposable (this is not true if two or more of the attached groups are identical, in which case certain spatial positions possess a superimposable mirror image).

[10] Attempts to make an optically active compound containing four different atoms, or groups, two atoms of which differ only in being isotopes of one another, *e. g.*, hydrogen and deuterium, up to now have not been successful. The differences between two such atoms apparently are not great enough to give a measurable result.

[11] For further discussion, see WHELAND, G. H., ref. 5, p. 168.

[12] Similarly, the number (and type) of isomers predicted for a pyramidal arrangement (where the carbon atom is at the apex of a pyramid, and the four valencies are directed toward the corners of the square base), or for any other spatial arrangement, does not agree with the isomers that actually do occur.

to four different groups, one predicts three stereoisomers, and specifically three *geometric* isomers. It will be shown later that such isomers are devoid of any (overall) optical activity because of the presence of a plane of symmetry.

MULTIPLE BONDS

For the purposes of this paper, the stereochemical effect of a carbon-carbon double bond is illustrated in Figure 3, which represents the hypothetical formation

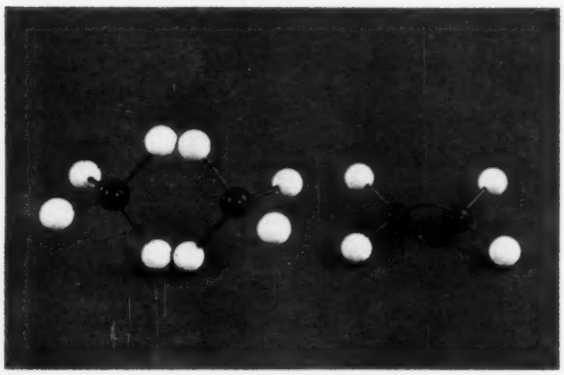


Figure 3

of ethylene from two molecules of methane (with elimination of hydrogen). It is evident that the double bond is responsible for the planarity of the ethylene molecule. Similar arguments apply to the carbon-nitrogen, carbon-oxygen, and other double bonds.[13]

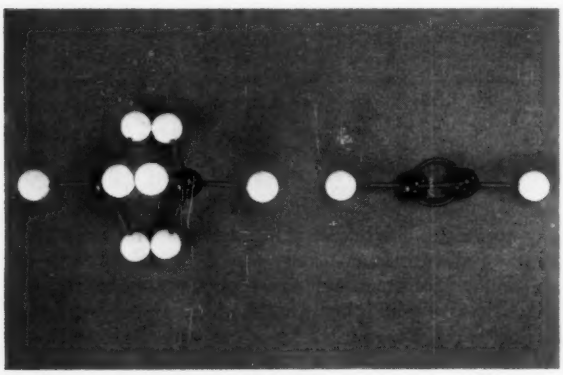


Figure 4

The formation of acetylene, by the hypothetical combination of two molecules of methane, is illustrated in the classical manner in Figure 4. It is evident that a carbon-carbon triple bond causes the molecule to become linear.[14]

ENANTIOMORPHISM

With the exception of one important physical property, namely, optical activity or the ability to rotate the plane of polarized light, all of the common physical and chemical properties of enantiomorphic isomers (such as melting points, boiling points, reaction rates, etc.) are identical (the physiological properties, however, may not be). Enantiomorphs differ in that they rotate the plane of polarized light to an equal but opposite extent.[15] The common physical and chemical properties of enantiomorphs are identical, however, only if (*a*) they are not mixed together, or (*b*) they are not reacted individually with a third compound which is itself optically active. When mixed, the properties of enantiomorphs are not identical with the properties of each isomer considered separately, but are dependent upon the composition of the mixture. Most of the properties of enantiomorphs are identical, of course, because of the great similarity to each other both structurally and spatially (*cf.* Structure III*a* with III*b*).

Optical activity is restricted to two classes of substances: (1) Those compounds whose molecules possess an *asymmetric arrangement of atoms within the molecule.* Such compounds are capable of displaying optical activity not only in the crystalline state but also in the liquid state or when in solution, for the individual molecules are optically active, each molecule possessing optical activity of the same sign at all times (though to a different degree at any particular instant). (2) Certain crystalline substances which, although not possessing an asymmetric arrangement of atoms in a molecule, possess an asymmetric arrangement of molecules, or atoms, throughout the whole crystal. Once the crystalline structure has been broken down by melting, solution, etc., the (overall) optical activity disappears.

As previously stated, an asymmetric molecule (whatever its particular "type" of asymmetry) by definition always possesses a noncoincident mirror image (in all of its spatial forms). In a large number of cases, it is the presence of an asymmetric carbon atom (or atoms) that gives the molecule as a whole its asymmetry and that causes the isomer to display (overall) optical activity. Nevertheless, the presence of such an atom is not a necessary condition for optical activity, for asymmetric molecules exist, *e. g.*, certain biphenyl derivatives, which do not possess such a carbon atom, and such molecules give rise to enantiomorphic isomers.

[13] According to classical theory, both the carbon-carbon bonds in ethylene are identical and the hydrogen-carbon-hydrogen bond angle should have approximately the tetrahedral value of 109°. According to molecular-orbital theory, however, one of the bonds (the so-called π bond) is different from the ordinary (or δ) single bond and is actually the weaker bond. In addition, the hydrogen-carbon-hydrogen bond angle is calculated to have the trigonal value of 120° (see ref. 3).

[14] Although modern theory also predicts a linear molecule for acetylene, it shows that the triple bond is made up of one strong δ bond and two weaker π bonds (see ref. 3).

[15] All statements concerning the optical activity of a compound refer to a statistical activity of an appreciable number of molecules and not to an individual molecule. As explained later, the optical activity of an individual molecule, in the great majority of compounds, varies from instant to instant

The relationship between two enantiomorphic molecules is like that between a right and a left hand; both hands have a similar structure, *i. e.*, contain five similar fingers, and each finger is connected in a similar manner. Thus, both thumbs are connected to the forefingers, and the latter are connected to the middle fingers, and so forth. But yet the actual spatial arrangements of the fingers in each hand are not identical and they exhibit a noncoincident, or nonsuperimposable, mirror-image relationship (when placed palm to palm, they are not being superimposed).

It is to be noted that the two enantiomorphic molecules, III*a* and III*b*, differ in one important respect; in III*a* there is a clockwise relationship existing between the atoms, or groups, B, X, and Y, while in III*b* there is a counterclockwise relationship (looking up underneath B, X, and Y). It must be stressed that even today we do not know with complete certainty which one of the two is responsible for the dextro rotation. In fact, until recent years, there were no known methods of identifying the absolute configuration of such a compound, and therefore one of the isomers was arbitrarily designated to be the dextro; its mirror image by definition, possessed the levo configuration.[16] Once the configuration of a compound containing only one asymmetric carbon had been established by decree, then the (relative) configurations of the more complicated molecules derived from it were based accordingly upon the arbitrary standard and could be determined experimentally, although this was not always easy.

How can one predict whether an appreciable number of molecules of a certain compound will be optically active? One method is to construct a molecular model of the molecule in question and of its mirror image and test for coincidence in all possible spatial positions; or we can represent the molecule and its mirror image on paper by means of spatial formulas. If the two models, or spatial formulas, are found to be nonsuperimposable at all times, the molecules they represent will be optically active in all spatial positions. The optical activity of an appreciable number of such molecules from the two (enantiomorphic) isomers is always equal and opposite.

A quicker method is to examine the molecule for the presence, or absence, of certain elements of symmetry. If all three of the following elements of symmetry (or rather, classes of elements) are absent from a molecule, an appreciable number of such molecules will always display overall activity: (*a*) a plane of symmetry, (*b*) a center, or point, of symmetry, and (*c*) an alternating axis of symmetry. As mentioned before, the alternating axis must be carefully distinguished from the *ordinary* axis (or line) of symmetry. Many asymmetric molecules do not possess an ordinary axis of symmetry, but some do and so its absence is not a necessary condition for overall optical activity. Therefore, this particular element of symmetry need never be considered in stereochemical problems.

It also should be mentioned that in practice it is only necessary to consider whether a molecule possesses either a *plane* or a *point* of symmetry. Theoretically, however, if neither of these two elements is present, the molecule would then be examined for an *alternating* axis of symmetry, because even if this were present alone, the compound would be inactive (*i. e.*, statistically). Although an alternating axis is often present in addition to the other elements of symmetry, so far no molecule possessing only an alternating axis has been prepared or found in nature, although one can readily enough "construct" one on paper.

It should be emphasized that a molecule is said to "possess" a particular element of symmetry, no matter how few spatial positions out of an infinite number possess such an element (*e. g.*, the biphenyl derivative, XIX*a*, described later, possesses a plane of symmetry in only *two* spatial positions, and yet this is sufficient to guarantee statistical inactivity). Each of the elements of symmetry will now be considered in detail.

Plane of Symmetry. A plane of symmetry is said be present in a molecule if the latter can be divided into two parts by an imaginary plane so that each half is the mirror image of the other half. Thus, the molecule, XI, has a plane of symmetry through the atoms (or groups) A, C, and the central carbon. Its mirror

(XI) (XII)

image, XII, is *identical* and therefore no enantiomorphism is possible. Whenever the central carbon is attached to four different groups (rather than three in XI), however, the molecule does possess a nonsuperimposable mirror image, because there can be no plane of symmetry present in such a molecule (or any other element of symmetry).

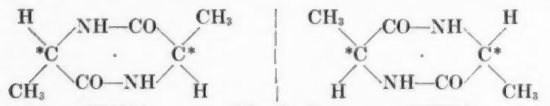
In order to divide the molecule into symmetrical halves, the dividing plane may or may not cut through atoms or groups. Ethane, for example (in certain spatial positions) possesses a plane of symmetry which cuts across the carbon-carbon bond; two of the five possible plans of symmetry in cyclobutane also cut across connecting bonds.

Point of Symmetry. A molecule is said to possess a point, or center, of symmetry if a point can be placed within the molecule so that when a line is drawn from any atom or group to this point and then extended, it will always meet a similar atom or group (at the same distance).[17] Thus, although *trans*-dimethyl-diketo-pi-

[16] For discussions concerning the possibility of determining absolute configurations, see PEERDMAN, A. F., A. J. VAN BOMMEL, AND J. M. BIJUOET, *Proc. Koninkl. Nederland. Akad. Wetenschap.*, **54B**, 16 (1951). See also WOOD, W. W., W. FICKETT, AND J. G. KIRKWOOD, *J. Chem. Phys.*, **20**, 561 (1952). For a recent review, see KUHN, W., *Z. Elektrochem.*, **56**, 506 (1952).

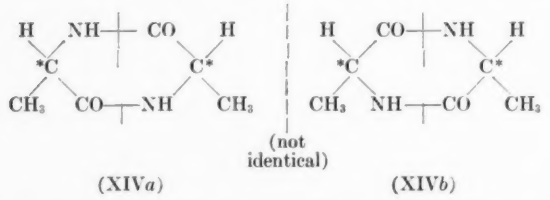
[17] When a point of symmetry is present in a molecule, there is always an alternating axis of symmetry present at the same time. A molecule can, however, possess an alternating axis without necessarily possessing a point of symmetry (at least in theory).

perazine, XIII*a*, does not possess a plane of symmetry, it does possess a point of symmetry, and its mirror image, XIII*b*, can be made to coincide with it. That



the two molecules are identical can be readily seen by inverting either molecule. For simplicity, the exact spatial relationships of every atom are not shown in the diagram. The methyl groups and hydrogen atoms attached to the asymmetric carbon atoms extend above and below the plane of the ring (not vertically but at the tetrahedral angle). The doubly bonded oxygen atoms are in the plane of the ring but stick out at an angle away from the ring (for further remarks concerning the piperazine ring, see the following section). It should be noted that there are two *similar* asymmetric carbon atoms present in the molecule; since it is optically inactive (*i. e.*, has no overall activity), it is also called a *meso* compound (see diastereoisomerism).

Axis of Symmetry. A molecule is said to possess an ordinary axis of symmetry if a line can be drawn through the molecule so that on rotating the latter about this line (or axis) through 360° it will coincide with itself at least twice. For example, let us consider *cis*-dimethyl-diketo-piperazine, XIV*a*. All three of the

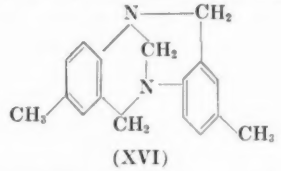


necessary elements of symmetry are absent in this molecule and therefore it possesses a noncoincident enantiomorphic partner, XIV*b*. However, the molecule does possess an ordinary axis of symmetry. As stated before, one never need worry about this element of symmetry because it has no significance to the organic chemist (at least in relation to stereoisomerism).

The planar form of the six-membered piperazine ring has been drawn for convenience, even though the ring system is undoubtedly puckered and comparatively strainless. But since presumably there is a dynamic equilibrium among the various forms (as in cyclohexane), including also the "intermediate" planar form, the possible number of isomers can be calculated on the basis of the planar formula. This would not be the case if the various geometric forms were capable of independent existence, for then the number of optical isomers would be greater.

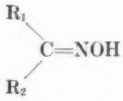
Although three different groups are attached to the nitrogen atoms in both XIII and XIV, they are not asymmetric in the same sense that the carbon atoms are, for a nitrogen substituted in this manner gives rise to no enantiomorphism of its own. A compound of the type NABC theoretically should give rise to two optical isomers, XV*a* and XV*b*. However, every

attempt to prepare an optically active derivative of ammonia containing a single trivalent nitrogen atom has failed so far. This is almost certainly due to an oscillation of the nitrogen atom which converts one optical isomer into the other, the oscillation being so rapid that no isolation of the two forms is possible. It is of interest to note, however, that one compound, Tröger's base, XVI, containing *two* asymmetric tri-



valent nitrogen atoms, has been resolved into optically active forms.[18] In this compound the nitrogen atoms are part of a complex (and more or less rigid) ring system, and are therefore prevented from vibrating to their usual extent.

When the nitrogen is linked by a double bond, separable isomers are possible. The isomerism exhibited by the oximes, for example, is very similar to that shown by certain derivatives of ethylene, *e. g.*, *sym*-dichloroethylene. Thus,



exists in two stereoisomeric forms, as shown by the configurational formulas XVII*a* and XVII*b*. The two forms have distinct physical and chemical properties,

are called *geometric* isomers. The central carbon and nitrogen atoms, and the three single bonds connected to the atoms, are all in the same plane (drawn here in the plane of the paper). The double bond can be picture as extending vertically above and below the plane of the paper. The isomerism is possible because of the lack of freedom of rotation about the carbon-nitrogen double bond (as with a carbon-carbon double bond).

Alternating Axis of Symmetry. A molecule is said to possess an *alternating* axis of symmetry if a line, or

[18] See PRELOG, V., AND P. WIELAND, *Helv. Chim. Acta*, **27**, 1127 (1944).

axis, can be drawn perpendicular to a plane dividing the molecule into two halves, which, on rotation of *one-half* of the molecule about that axis (in either direction), will cause that half to become at certain intervals the mirror image of the opposite half. (If necessary, the imaginary plane can, of course, cut symmetrically through atoms or groups.) It is called an alternating axis because rotation of one-half of the molecule will cause that half to become alternately "coincident" and "noncoincident" (in regard to a mirror-image relationship) with the opposite half.

The piperazine derivative, XIII, for example, possesses an alternating axis of symmetry (in addition to a point of symmetry). As stated previously, in practice this element of symmetry is not important, and so no description will be given here of a (hypothetical) molecule which has none of the decisive elements of symmetry present except for the alternating axis.[19]

FREEDOM OF ROTATION

The so-called freedom of rotation about a single bond does not mean *complete* freedom. Even in the ethane molecule, there is some interference between the hydrogen atoms in the two methyl groups. This means that the groups will not rotate about each other until a certain minimum force has been applied. The forces causing rotation are, in general, the impacts of the molecules themselves, and usually these impacts will be sufficiently powerful to bring about rotation. Although it appears that by no means all of the impacts are able to cause rotation, the latter will occur frequently enough so that from the standpoint of the chemist it can be considered to occur in a continuous fashion.[20]

Quantum theory has shown, in addition, that the methyl groups cannot possess *any* value of rotational energy, *i. e.*, a continuous series of energy values is not possible. The methyl groups can only possess certain discrete multiples of the minimum possible energy (the rotational energy of the molecule as a whole is, of course, also quantized).

During rotation of the methyl groups in ethane, all the possible spatial positions are not equally probable. Ignoring collisions, etc., the methyl groups possess a constant (total) energy, but they do *not* possess a constant velocity. The potential energy of the groups varies during rotation, owing to differences in the attractive and repulsive forces of the atoms in the different spatial positions, and therefore the kinetic energy and velocity of these groups will also vary. These remarks, of course, apply equally well to all molecules possessing groups in rotation about a single bond.[21]

[19] For a description of such a molecule, see SHRINER, R. L., R. ADAMS, AND C. S. MARVEL, in GILMAN'S "Organic Chemistry," 2nd ed., John Wiley & Sons, Inc., New York, **1943**, Vol. 1, p. 319.

[20] See RICE, O. K., "Electronic Structure and Chemical Binding," McGraw-Hill Book Co., Inc., New York, **1940**, p. 273. See also PAULING, L., "The Nature of the Chemical Bond," 2nd ed., Cornell University Press, Ithaca, N. Y., **1940**, pp. 90–2.

[21] For further discussion, see WHELAND, G. W., ref. 5, pp. 175–80.

MOLECULAR AND STATISTICAL ACTIVITY[22]

Let us first consider substances displaying (overall) optical inactivity. In the majority of organic molecules belonging to this class, there are, of course, various groups present which are in rotation (or rotation-oscillation) about single bonds. Because of this there are frequently a large number of (transitory) spatial positions in such molecules which do not possess a plane or a point of symmetry. Whenever a molecule is in such a configuration, it is optically *active. If, however, the molecule is at any time capable of possessing a plane or point of symmetry, the overall optical activity displayed by an appreciable number of such molecules is always found to be zero.* In such cases, the optical activity displayed by one molecule in a particular asymmetric configuration is always (found to be) balanced by the presence of another molecule possessing a mirror-image configuration and therefore of opposite activity (this is simply a result of the symmetry of such molecules).

Let us now consider the optically active substances. *If a molecule is at no time capable of possessing a plane or point of symmetry, the overall optical activity of an appreciable number of such molecules is not zero.* In such cases, the molecule rotates the plane of polarized light in the *same* direction (though to a different degree) in all possible spatial positions, *i. e.*, configurations which are mirror images of one another do not occur in such molecules.

Questions concerning the optical activity of an individual molecule are, of course, somewhat speculative since experimental results can only be obtained from a considerable number of molecules. However, it appears reasonably certain that the following types of molecules are completely inactive at all times and no matter in what direction the beam of polarized light approaches the molecule: (1) Those molecules which possess a fixed configuration of atoms and a point of symmetry, *e. g.*, ethylene, benzene, cyclobutane, etc. Some molecules of course, because of freedom of rotation, only possess a point of symmetry when the atoms are in certain positions, so that the molecule may be optically active in many of the other spatial forms. (2) Those molecules which, although not possessing a point of symmetry, nevertheless possess an extremely high degree of symmetry, *e. g.*, carbon tetrachloride (the molecule does, of course, possess a plane of symmetry).

Let us now consider a molecule like chloroform, possessing a fixed configuration and a plane of symmetry, which is not in either class (1) or (2). If a beam of plane-polarized light passes through the molecule so that it is perpendicular to the plane of symmetry, it can be shown that the resulting rotation is zero. It also appears likely that the rotation is zero if the plane

[22] The present discussion is largely based on articles by NOLLER, C. R., J. CHEM. EDUC., **24**, 600 (1947); MOWERY, D. F., *ibid.*, **29**, 138 (1952); WHELAND, G. W., ref. 5, pp. 130–47, 190–2; NOLLER, C. R., "Chemistry of Organic Compounds," W. B. Saunders and Co., Philadelphia, **1951**, p. 341. See also FREUDENBERG, K., "Stereochemie," Franz Deuticke, Leipzig, **1933**, lithoprinted by Edwards Brothers, Inc., Ann Arbor, Mich., **1945**, pp. 317 ff.

of symmetry is parallel to the plane of polarization. However, in all other orientations, the molecule is optically active. The overall activity of a large number of molecules of chloroform is zero because the probability of a particular molecule rotating the plane in a dextro manner is exactly equal to the probability of it rotating it in a levo manner, and so the individual activities of the various molecules cancel out by external compensation.

Let us now examine ethyl bromide, a molecule containing groups which are capable of freedom of rotation. It will be shown that out of an infinite number of possible spatial positions only six configurations possess a plane of symmetry. In all other configurations, the molecule is asymmetric and optically active (in whatever direction the beam of polarized light approaches the molecule). Nevertheless, as in the previous case statistical inactivity results.

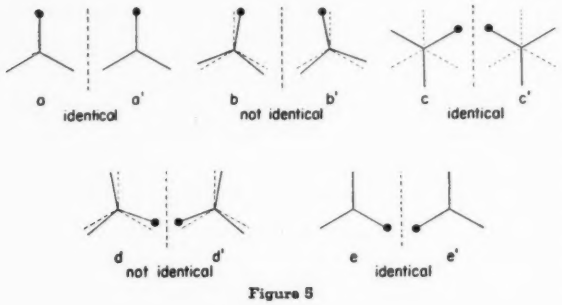


Figure 5

Let us suppose that the molecule is being observed directly above the axis of rotation (of the two methyl groups) and that the group containing the bromine atom is nearest to us (represented in Figure 5 by unbroken lines, the dot representing the bromine). Let us examine the different spatial arrangements that occur during one third of a rotation. In position *a*, the atoms in the CH_2Br group are pictured as being directly above the hydrogen atoms in the unsubstituted methyl. After one-third of a rotation, the former are again directly above the hydrogen atoms, as shown in position *e*. The molecule in both positions *a* and *e* possesses a superimposable mirror image and is optically inactive (in certain directions with respect to the polarized light), for it possesses a plane of symmetry in these positions.

Similarly, after one-sixth of a rotation, the molecule is optically inactive (in certain directions), as shown in position *c*. However, let us examine an intermediate position, *b*, for example. The molecule is now optically active (in all directions with respect to the beam of light) because it does not possess any of the necessary elements of symmetry. In fact, theoretically, this is true for all spatial positions that are possible between those represented by *a* and *c*.

The degree of optical activity will depend (in part) on the "degree" of asymmetry possessed by the molecule. Thus, as the spatial positions of the groups change from *a* to *c*, the molecule exhibits greater and greater asymmetry and, therefore, presumably is capable of exhibiting greater and greater optical activity, until a maximum is reached when the groups are in a position exactly halfway between *a* and *c* (*i. e.*, until one-twelfth of a rotation has occurred). After this point has been reached, the molecule exhibits less and less asymmetry and optical activity, until the latter reaches zero at position *c*.

A similar situation occurs between positions *c* and *e*. However, the optical activity is now reversed in these positions, because each one of these is the mirror image of a corresponding position between *a* and *c*. Thus *d* and *b* (which represent "corresponding" intermediate positions) are mirror images of each other, *i. e.*, *d* is identical with *b'*. Although all possible dextro or levo positions are not equally probable, it is true that when each dextro position is compared to the corresponding levo position, each pair of positions is equally probable. Thus, positions *d* and *b* are equally probable (this is true for ethyl bromide because the bottom methyl group contains three identical atoms).

If we consider a molecule of the type, CH_3CXYZ, however, the situation is different because of the presence of a single asymmetric carbon atom. By examining a molecular model of either of the isomers, it can readily be seen that the molecule is asymmetric, and therefore optically active at all times. In addition, at no time will certain positions be the mirror images of others, *i. e.*, the molecule does not pass through both dextro or levo configurations, so that the molecules of one isomer will *all* be either dextro- or levo-rotatory. The same type of argument applies, of course, to any of the more complicated molecules.

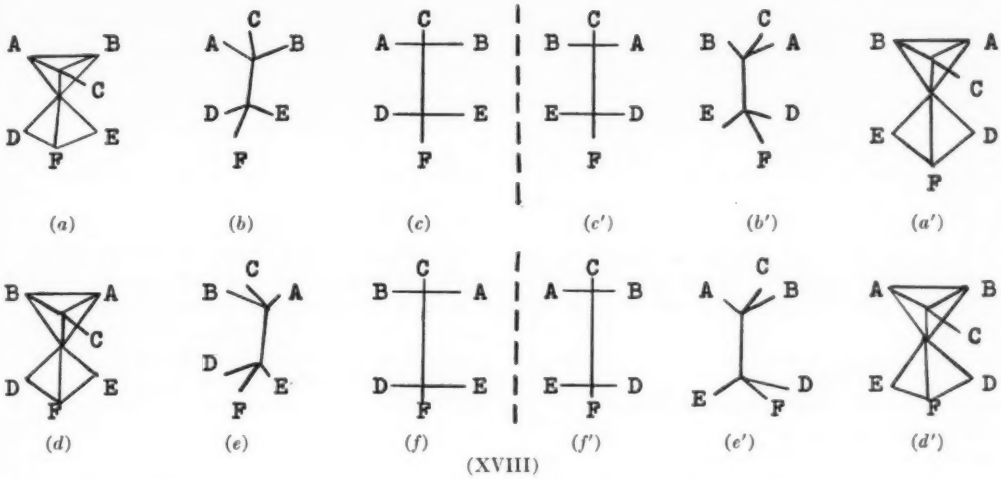
ENANTIOMORPHISM (*Continued*)

The following statements can be made concerning compounds with one or more asymmetric carbon atoms (which can always be checked by actual construction of molecular models). Once more it should be kept in mind that each isomeric "form" consists of an infinite number of different spatial forms. However, if there is, for example, a clockwise relationship existing between certain atoms or groups in one of the isomers, this relationship persists during rotation; also a counterclockwise relationship exists, and persists during rotation, in its enantiomorphic, or mirror-image form.

(1) Any compound, whether "straight" -chain, cyclic, fused-cyclic or any combination of these, which contains a *single* carbon connected to four *different* groups (*i. e.*, which contains a single asymmetric carbon atom) always possesses nonsuperimposable mirror image, and is therefore capable of existing in *two* spatial forms which exhibit an enantiomorphic relationship to one another.

(2) Any "straight" -chain or *mono*cyclic compound possessing *n different* asymmetric carbon atoms will be capable of existing in 2^n enantiomorphic modifications, or $2^n/2$ pairs of optical isomers.

It is to be noted that this general formula does not always apply to certain fused-ring compounds contain-



(XVIII)

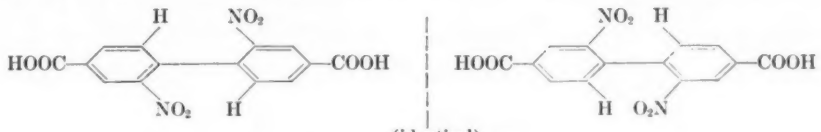
ing two or more dissimilar asymmetric atoms; the number of possible isomers may be less than 2^n in such compounds because of steric factors. In addition, it does not apply to *any* compound possessing *similar* asymmetric carbon atoms, the total number of possible isomers always being less in these cases (see discussion of diastereoisomerism).

Only one example will be considered here, namely, a fully substituted ethane molecule containing two different asymmetric carbon atoms.[23] On substituting in the general formula, it can be seen that four (2^2) different stereoisomers, *i. e.*, two enantiomorphic pairs, should be possible. Using the tetrahedral, valence-bond, and projection formulas, the first pair of enantiomorphs, exhibiting a mirror-image relationship to each other, is represented by XVIII*a* (*b* and *c*) and XVIII*a*′ (*b*′ and *c*′), and the second pair by XVIII*d* (*e* and *f*) and XVIII*d*′ (*e*′ and *f*′).

It is to be noted that on looking down on "top of" the molecule, XVIII*a*, that there is a clockwise relationship between the groups A, B, and C, taken in that order; similarly, looking up at the "bottom of" XVIII*a*, there is a counterclockwise relationship existing between the groups D, E, and F. These clockwise or counterclockwise relationships are exactly opposite in the enantiomorphic partner, XVIII*a*′. Similar relationships are seen to exist between the second pair of enantiomorphs, XVIII*d* and *d*′.

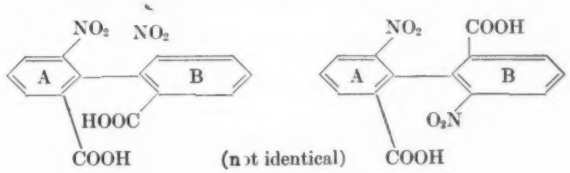
Enantiomorphism without Asymmetric Carbon Atoms. The *basic* requirement for enantiomorphism is that the molecule must be (at all times) *asymmetric as a whole* for, by definition, such a molecule always possesses a nonsuperimposable image. Although the majority of optically active compounds contain asymmetric carbon atoms, there are a number of compounds, *e. g.*, certain allenes, spiranes, biphenyls, etc., that do not.[24] Only one example of the biphenyl type will be given here.

Let us examine the biphenyl derivative, XIX*a*. The atoms and groups in the ortho positions are unable to interfere with one another very much even when the two rings are coplanar (as drawn in XIX*a*). The molecule possesses a plane of symmetry when the two rings are coplanar, and therefore no enantiomorphism is possible.[25]



(XIX*a*) (XIX*b*)

A molecule such as XX*a*, however, possesses such



(XX*a*) (XX*b*)

[23] For the more complicated cases, *e. g.*, those compounds which contain three asymmetric carbon atoms, two of which are the same, etc., see SCHRINER, R. L., *et al.*, ref. 19, pp. 234–40.

[24] For an extensive treatment of such compounds, see SHRINER, R. L., *et al.*, ref. 19, pp. 336–82. See also WHELAND, G. W., ref. 5, pp. 198–217.

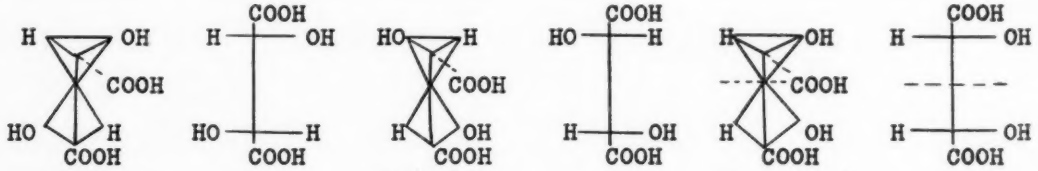
[25] In all other spatial positions except the coplanar position, the molecule does not possess a plane of symmetry and is optically active. As explained previously, however, the overall activity of an appreciable number of molecules is zero because of a statistical balance between equal numbers of dextro and levo molecules.

bulky ortho groups that a coplanar position is not possible, *i. e.*, complete rotation cannot occur but only rotation-oscillation. There are two possibilities for rotation-oscillation. If ring A is represented in the plane of the paper, the nitro group in ring B can be either above or below this plane. In XX*a* it is represented as being below and in XX*b* as above. For every possible spatial position, during rotation-oscillation of XX*a*, a corresponding nonsuperimposable, mirror-image form is possible (and equally probable) in the molecule represented by XX*b*. Thus, XX*a* and XX*b* represent optically active, or enantiomorphic, modifications.

DIASTEREOISOMERISM

Diastereoisomerism, like enantiomorphism, is a branch of optical isomerism (see chart in Part II). There are three types of diastereoisomers. As previously described, a nonsuperimposable, mirror-image relationship exists between any two enantiomorphic molecules. However, when two (stereoisomeric) molecules are said to be diastereoisomers of one another, there is no such mirror-image relationship, although such molecules are also nonsuperimposable.

Type (*A*). *Meso-Compounds.* Only those molecules that possess two or more *similar* asymmetric carbon atoms are capable of possessing meso stereoisomeric forms. Tartaric acid, for example, containing two similar asymmetric atoms, is capable of existing in three different spatial forms, as shown in XXI.



(XXI)

Meso-tartaric acid, XXI*c*, is called a diastereoisomer of both *d*-tartaric acid and *l*-tartaric acid, and it differs from them in both chemical and physical properties. In XXI*a*, there is a clockwise relationship existing between the groups, H, OH, and COOH, taken in that order, in both of the tetrahedra (looking down at the top tetrahedron, and up at the bottom tetrahedron), while in XXI*b* (its enantiomorph) there is a counterclockwise relationship. In meso-tartaric acid, XXI*c*, there exists a clockwise relationship in one-half and a counterclockwise relationship in the other.

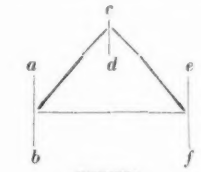
Classically, it was stated that meso-tartaric acid was optically inactive because of internal compensation, the activity of each half supposedly canceling out each other. It is true that in the position shown, XXI*c*, the molecule does possess a plane of symmetry and is therefore optically inactive (in certain directions with respect to polarized light). But, because of the freedom of rotation, in every other position—except for one where it possesses a point of symmetry[26]—it is optically active. As shown previously with other molecules, the optical activity of a "practical" sample of meso-tartaric acid is zero because of a statistical balance between equal numbers of dextro and levo forms. In other words, the so-called internally compensated compounds are actually, to a great extent, *externally* compensated.

Type (*B*). This includes those optical isomers that rotate the plane of polarization to a different degree in either the same direction or in an opposite direction. Referring to the molecules represented in XVIII, we can say that the relationship between *a* and *a′*, or between *d* and *d′*, is enantiomorphic. The relationship between *a* and *d* or between *a* and *d′*, for example, is said to be diastereoisomeric. They are optical isomers of one another but they do not display a mirror-image relationship.

Type (*C*). This includes certain types of ionic compounds (see chart in Part II).

GEOMETRIC OR CIS-TRANS ISOMERISM[27]

Only a few of the various types of geometric isomers will be considered here.[28] Although there is no difficulty in understanding geometric isomerism, there is, unfortunately, considerable difficulty in defining it. As a result, it is usually described as that type of stereoisomerism which is dependent upon some form of restricted rotation. This definition is fairly satisfactory in regard to the aliphatic compounds, but it is a poor one when applied to certain cyclic compounds, for a number of reasons. Thus, it is too broad in its scope,



(XXII)

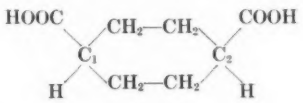
[26] The molecule possesses a point of symmetry if the top half of the molecule in position XXI*c* is rotated through 180°, the point being located at the junction of the two tetrahedra.

[27] The term *cis-trans* is usually reserved for certain of the simpler types of stereoisomers, *e. g.*, *cis-* and *trans*-dichloroethylene. In the more complicated cases, *e. g.*, XXII, the term obviously cannot have any meaning in regard to the molecule *as a whole* but only with reference to specific groups.

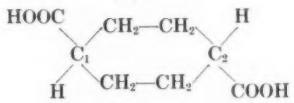
[28] For a thorough discussion, see SHRINER, R. L., *et al.*, ref. 19, pp. 444–87.

because restricted rotation can also bring about optical isomerism, *e. g.*, in the biphenyl compounds. Secondly, as pointed out by Wheland, although restricted rotation is present in the cyclic compounds, stereoisomerism would still occur even if freedom of rotation were present.[29]

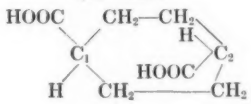
Let us consider cyclohexane 1,4-dicarboxylic acid, for example, which exists in two spatially isomeric forms, as shown in XXIII*a* and XXIII*b*. A "simple" rotation of the groups on C_2 would not (as commonly



(*a*) *cis*



(*b*) *trans*



(*c*)

(XXIII)

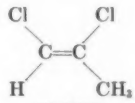
described) convert *a* into *b*. The rotation would (if it were possible) give rise to *c*. Only if the groups attached to C_2 were at the same time turned inside out would the rotation convert *a* into *b*. This is certainly not what is usually meant by freedom of rotation.

In the opinion of the writer, it would be more satisfactory to limit the meaning of the term *geometric isomer*, and to *classify as geometric only those spatial isomers which cannot otherwise be classified as optical isomers*. Stereoisomerism could then be divided into two nonoverlapping branches, (*a*) optical isomerism (encompassing enantiomorphism and diastereoisomerism) and (*b*) geometric isomerism, which would include all other spatial isomers.

Admittedly, a "negative" classification such as this would mean including a fairly "heterogeneous" group of compounds, but it would, nevertheless, avoid unnecessary duplication. In practice, only those molecules which satisfied the following conditions would be classified as geometric: (1) The molecules must be capable of possessing different spatial arrangements of atoms. (2) They must not contain *any* asymmetric carbon atoms. (3) The molecules must not be *asymmetric as a whole* (*i. e.*, if freedom of rotation is present, they must possess, at some time or other, a plane or point of symmetry).

Condition (2) would exclude enantiomorphs containing *dissimilar* asymmetric atoms and also the meso compounds containing *similar* asymmetric atoms. Condition (3) would exclude all other enantiomorphs. Meso compounds would, therefore, always be classified as *optical* isomers and never as geometric (contrary to present-day practice in the case of the cyclic compounds, as will be shown).

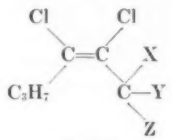
It should be remembered that although an appreciable number of molecules of a geometric isomer (by the above definition) do not display any overall optical activity, the majority of individual molecules often do display it. Thus, the methyl derivative of *cis*-dichloroethylene, XXIV, for example, is theoretically



(XXIV)

optically *active* most of the time during rotation of the methyl group (the molecule possesses a plane of symmetry only in a few spatial positions). Once again, it is a question of *statistical* inactivity.

It is, of course, always possible for different types of isomerism (optical, geometric, or structural) to be present in a molecule at the same time. The double bond in XXV, for example, is capable of giving rise to



(XXV)

geometric isomerism. The propyl group, on the other hand, can give rise to *structural* isomerism, for it can either be normal or branched. The group —CXYZ causes *optical* isomerism. No matter how we define our terms, complicating factors such as these can always occur.

CYCLOPROPANE ISOMERISM

The stereoisomerism exhibited by the cyclopropane molecule containing one or more different asymmetric carbon atoms is similar to that of the aliphatic compounds in that the number of stereoisomers can be calculated using the general formula, 2^n. A molecule of the type, XXVI*a*, containing one asymmetric carbon atom, possesses a nonsuperimposable image, XXVI*b*,

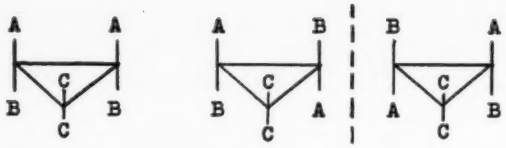
(*a*) (*b*)

(XXVI)

and is therefore capable of existing in two enantiomorphic forms. If two different asymmetric atoms are present, then four stereoisomers are possible.

When two similar asymmetric atoms are present, three stereoisomers are possible. The meso form,

[29] See WHELAND, G. W., ref. 5, pp. 194–8 and 217–23.



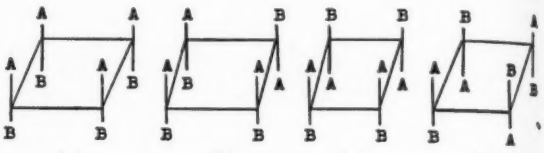
(*a*) meso (*b*) (*c*) (racemic pair)

(XXVII)

XXVII*a*, possesses a plane of symmetry and is optically inactive, like its aliphatic analogue. The pair of enantiomorphs is represented by XXVII*b* and *c*. Now, according to the usual definition, XXVII*a* is said to be a *geometric* isomer of either *b* or *c*. This is somewhat illogical, for the meso forms in the aliphatic series are never called geometric.

According to our restricted definition, the meso form would simply be a *diastereoisomer* of either *b* or *c*, the term geometric being omitted altogether. Admittedly, the term geometric was applied to such isomers because in XXVII*a* the groups, A and B, are on the same side of the molecule (which was therefore called the *cis*-isomer), while in *b* and *c* they are on opposite sides (the *trans*-modification giving rise to two enantiomorphic forms). The properties exhibited by *a* are, of course, different from those of *b* and *c*. However, this is true of all diastereoisomers.

Finally, let us consider a cyclobutane derivative of the type, XXVIII. By altering the way in which the groups are arranged above and below the plane, it can



(*a*) (*b*) (*c*) (*d*)

(XXVIII)

be seen that only four isomers (XXVIII*a–d*) are possible. There are no asymmetric carbon atoms present in the molecule and, in addition, none of the molecules are asymmetric as a whole (they all possess a plane, or planes, of symmetry). All four isomers are, therefore, geometric. Similar reasoning can be applied to the more complicated molecules.

ACKNOWLEDGMENT

The author wishes to thank G. W. Wheland, of the University of Chicago, and B. O'Brien, of the University of Rochester, for valuable advice concerning optical activity, and also M. L. Huggins and J. R. Thirtle, of these Laboratories, for critically examining the manuscript and for many helpful remarks.

STUDENT EXPERIMENTS IN INFRARED SPECTROSCOPY

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THE increasing use of the infrared spectrometer as an analytical tool makes it highly desirable for the graduate chemistry student to become familiar with this instrument. Accordingly we have introduced an experiment in infrared spectroscopy as part of a graduate course in instrumental analysis. Since extensive experience is required before one can really master the use of the instrument, we have tried only to give the student some idea of the problems which can be solved by infrared spectroscopy and the general experimental methods which are used. The two problems which we have chosen are the qualitative identification of characteristic structural features of molecules and the quantitative analysis of a binary mixture.

OPERATION

The first laboratory period is spent largely in familiarizing the students with the operation of our own particular instrument, a Perkin-Elmer model 12-C. Since the instruction manual is not able to keep pace with the changes in instrument design, it has proved easiest for an experienced spectroscopist personally to instruct the students in groups of three and four. This provides an opportunity to stress the careful handling of

the rock salt windows and cells and to include suggestions on the easiest ways of filling cells and handling samples. Usually the covers are removed from the spectrometer and the students are given the opportunity to see the optical system. After the instruction period the students are allowed to operate the instrument themselves under rather close supervision. Usually a sample is not used in this practice period, but the background spectrum of atmospheric absorption superimposed on the curve of source intensity is run. This acquaints the students with the spectral regions in which the background spectrum is particularly important and emphasizes the fact that this background cannot be ignored in infrared spectroscopy. The students are encouraged to experiment and see for themselves the effect on the spectrum when slit width and amplifier gain are varied.

DETERMINATION OF SPECTRA

The second laboratory period is devoted to the determination of the spectra of pure compounds and the interpretation of these spectra. Each pair of students is assigned two different compounds which comprise the binary mixture which they will analyze later. In selecting these, several criteria are set up:

1. The pure compounds should be readily available.
2. They should contain a variety of structural features which are discernible from their spectra.
3. The spectra of the compounds should be available in the literature for reference.
4. Each binary mixture should not be capable of easy analysis by a simpler method; in other words, the analytical problem given to the student should be a practical one.
5. For convenience, the compounds should be liquids.

Suitable pairs of liquids are benzene and toluene, cyclohexane and methyl cyclohexane, pairs of the isomeric butyl alcohols, benzene, and thiophene, and of course many others.

The spectrum of each pure compound is determined over the range from 2 to 15 μ. Suitable instrument settings and cell thicknesses are found by the process of trial and error. For substances such as hydrocarbons that do not exhibit intense absorption a cell thickness of 0.1 mm. is usually satisfactory. For other substances, such as alcohols, whose absorption is more intense a cell thickness of 0.025 mm. is generally appropriate. As an alternative procedure these latter substances may be diluted with carbon tetrachloride or carbon disulfide. However, if the spectra of two or more compounds are to be run under identical conditions in order to serve as a basis for working out a quantitative analysis, this dilution step introduces an extra manipulation which can be a source of error, particularly in the hands of an inexperienced student.

When satisfactory results have been obtained for the two samples and for the background, the student then examines the spectra in an attempt to correlate absorption frequencies with the structures of the samples. In order to do this the spectra obtained from an ordinary single-beam instrument must be replotted. First it is necessary to take the spectrum of a sample and the background spectrum and superimpose them on a light box. The ratio between the transmission of the sample and that of the background at the same wave length is calculated, and the ratios so obtained are plotted against wave length. For an instrument such as the Perkin-Elmer where the wave-length scale is not linear, a calibration chart must be used to convert wave-length dial readings to wave length. A fairly satisfactory spectrum may be obtained by accurately calculating ratios only at the transmission maxima and minima and then completing the plot by sketching in the bands. Even this type of plotting is a laborious task for an inexperienced student, and therefore we have found it advisable to eliminate some of this work by plotting only part of the spectrum—for instance from 2.5 to 10 μ. It may be argued that it is unnecessary to replot a spectrum in order to interpret it, but we feel that the student gains much by this experience. He becomes acquainted with the process which is necessary for preparing a spectrum suitable for publication. He is made aware of the different ways in which a spectrum may be plotted and realizes the deplorable lack of uniformity among spectra reported in the literature. He is able to compare the spectrum he has obtained with those reported in the literature and check on the accuracy of his work.

When the student has obtained a satisfactory replotted spectrum he is then referred to the literature (*1*, *2*, *3*, *4*) for correlations of absorption frequency and structure. We usually attempt to characterize the strong bands between 2.5 and 7.5 μ, and only in cases where the student is particularly interested do we attempt to characterize bands at longer wave lengths.

ANALYSIS OF MIXTURE

From one and one-half to two laboratory periods are devoted to the preparation of a working curve and the analysis of a binary mixture. Before the student begins work on a mixture he is required to read certain articles in the literature designed to acquaint him with the techniques involved and the errors which may occur. We assign the section on quantitative analysis in the Perkin-Elmer instruction manual (*5*) and the first of Robinson's papers on quantitative analysis (*6*). We make the assumption that a graduate student is already familiar with Beer's law and the fundamental principles of spectrophotometric analysis. In cases where the student's background is known to be inadequate, he is urged to review the material in any standard physical chemistry text and laboratory manual.

When the student has a reasonable grasp of quantitative methods of infrared spectroscopy he is then ready to turn to his own particular problem. We have the students work in pairs because of the limitations of time and equipment. As mentioned previously each pair of students will have determined the spectra of the components of their binary mixture. These spectra (the originals—not the replotted ones) are compared in order to select absorption bands suitable for analysis.

For the pairs of compounds which were listed earlier it is usually possible to find one strong band for each component at a wave length where the other component shows but little absorption.

The method of analysis which has been most successful in our case involves the preparation of a working curve in which the absorbence of a mixture is plotted as a function of the percentage of one of the components in the mixture. To construct this working curve three or four synthetic mixtures are prepared by combining accurately weighed amounts of the pure components. We cover the entire range of concentration by preparing mixtures containing 25, 50, and 75 per cent or 20, 40, 60, and 80 per cent of the compound to be determined. Keeping in mind the material he has read about errors in measurement and the effect of slit width, the student then determines the instrument settings which will yield satisfactory analytical results. There are two ways in which this may be done, and both will be discussed here. Each method involves measurement of a zero transmission, a 100 per cent transmission, and measurement of the transmission of the mixture.

Measurements Made by Using the Recorder. The zero setting is made first, and then with the least absorbing component in the cell several short scans are made of the region where the analysis is to be made, varying the amplifier gain and slit width until a transmission of approximately 100 per cent is obtained. Once the proper settings are found these values are recorded so that all subsequent measurements may be made under exactly the same conditions. The 100 per cent trace made under these conditions is saved for use in calculating I_o values later. Using the instrument settings determined above, short scans are made of each of the synthetic mixtures. The minimum transmission values (the peaks of the absorption bands) on each of the scans made on the mixture are used as I values. I_o values are obtained at the same wave length from the 100 per cent trace made earlier. The working curve is then constructed by plotting values of long I_o/I versus percentage of one of the components in the mixture.

Measurements Made by Using the Densitometer. In this method the zero setting is first made, and then with the least absorbing component in the cell and the wavelength dial set at the exact wave length to be used, the 100 per cent adjustment is made by varying the slit width and densitometer controls. (The student should realize that this wave-length dial setting may not be exactly the same as that indicated by the recorded spectrum because of the finite time required for the recorder response.) With the densitometer controls, slit width, and wave-length dial at exactly the correct settings, the absorbencies (optical densities) for each of the synthetic mixtures are read directly from the densitometer. The working curve is constructed by plotting these absorbencies against the percentage of one of the components in the mixture. In our experience, when the absorption bands used in the analysis are quite sharp it is best to check the wave-length dial setting very carefully to make sure that it is actually on the peak of the band. This is particularly important when the original wave-length setting has been made using a fairly concentrated solution giving relatively broad bands, and subsequent measurements are to be made on more dilute solutions giving sharper bands.

DISCUSSION

We have found that the average graduate student in his first attempt at using the infrared spectrometer for analysis does not obtain particularly reproducible or accurate results. The working curves plotted by our students usually obey Beer's law within experimental error; however, this error may be from 0.05 to 0.10 absorbency units. The results obtained using the recorder are usually more reproducible than those made using the densitometer. In the last analysis the accuracy of the results seems to be largely a function of the amount of time spent in repetition of the measurements. Whether or not the student actually analyzes an unknown mixture is left to the discretion of the instructor. In certain cases it may be felt that analysis of an unknown adds to the student's interest in the experiment; in other cases because of limitation of time this may be omitted. If the entire range of composition from 0 to 100 per cent is covered the student can probably not be expected to come closer than ± 5 per cent in the analysis of a mixture.

The results of this work have been most satisfactory to the students and to the instructor. In spite of the fact that the results of the analysis are not highly accurate, they are sufficiently good so that the student feels that the experiment has not been a failure. Usually the sources of error are quite obvious to the student even before they are pointed out by the instructor.

Originally we had certain misgivings about allowing so many students to use an instrument as delicate as an infrared spectrometer. We were particularly concerned about possible damage to sample cells valued at approximately $90. We have had absolutely no trouble resulting from careless handling of the equipment other than occasional difficulties with the string on the automatic slit drive. Fortunately, this trouble is easily corrected by anyone who has had a little experience in using the instrument. Actually, teaching the use of the spectrometer as a part of a course taken by most of the graduate students will probably avoid damage to the instrument which might result when a graduate student, eager to obtain results bearing on his own research problem, does not take sufficient time to learn to use the equipment properly.

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AN INEXPENSIVE, EASILY CONSTRUCTED SPECTROPHOTOMETER[1]

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MANUALLY operated spectrophotometers that are commercially available, and usable in the range of the visible spectrum, vary in price from approximately $400 to $1300. In certain of these instruments the monochromator is a diffraction grating, and the resulting spectral dispersion is linear throughout the wavelength range covered. Other spectrophotometers have quartz or glass prisms as spectral dispersing elements. Considered by some to be a disadvantage is the fact that prismatic dispersion is nonlinear.

In recent years interference filters have been developed for use in filter photometers and other optical instruments. Unlike glass or gelatin absorption filters which contain dissolved or suspended coloring agents, most of these newer filters isolate narrow spectral bands through interference of light waves reflected from two partially transparent, parallel, metallic layers separated by a thin spacer layer of transparent material. This principle underlies the operation of the Fabry-Perot interferometer.

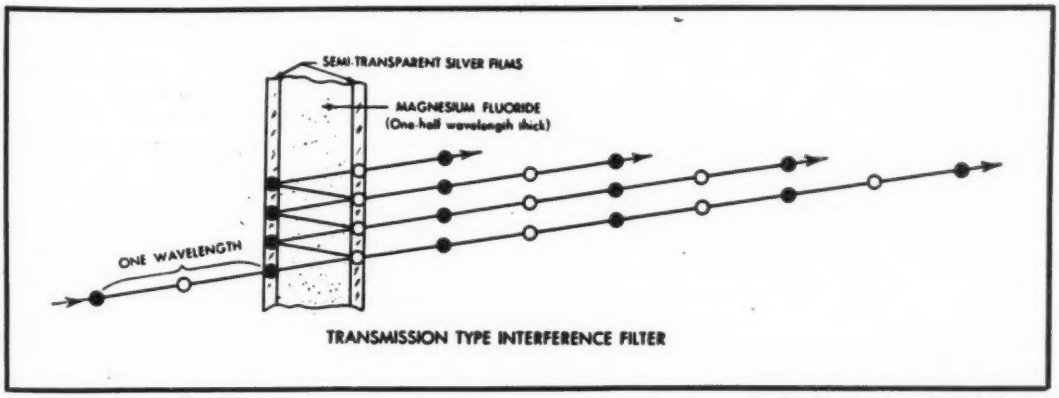
In Figure 1 the construction of a typical transmission-type interference filter is shown. Two semitransparent silver films are separated an extremely short distance by a spacer layer of low refractive index material, in this case, magnesium fluoride. In practice, cover glasses are added for protection.

Consider now the light ray at the lower ieft striking the first silver film. Part of this ray is transmitted through both the metallic layer and the magnesium fluoride layer to the second silver film. Here, part of the light ray is transmitted as shown, and part is reflected back to the first metallic layer. A part of this backward-reflected light is reflected forward again, and so on. Assuming that the incident light is monochromatic, the magnesium fluoride layer is represented as having a thickness of one-half a wave length. Thus, during each double reflection a light ray travels twice this distance, so that successive emergent rays are one wave length apart and reinforce one another. With heterogeneous white light as the incident beam, the various component wave lengths will have different path lengths and differ in phase to different extents, so that rays of some wave lengths will be almost extinguished while rays of other wave lengths will show maximum transmission.

In the interest of accuracy it should be pointed out that this drawing of an incident light ray at a slight angle from the perpendicular is done merely for illustrative purposes. When this ray is normal to the first surface, all lines shown merge into one and the parts passing the second metallic film become components of a single ray. However, with white light these components differ in phase and the resulting color of the transmitted light is determined by the thickness of the magnesium fluoride layer. Interference filters of this sort are available to cover a spectral range from about 400 to 900 mμ.

Recently from the Bausch & Lomb Optical Company experimental wedge interference filters have been made available (*1*). In these, the transparent spacer material is made in the form of a linear wedge, that is, the thickness of the spacer layer varies uniformly from one end to the other. Thus from one end to the other,

[1] Presented at the 122nd Meeting of the American Chemical Society, Atlantic City, September, 1952.



Courtesy Bausch & Lomb Optical Company

Figure 1

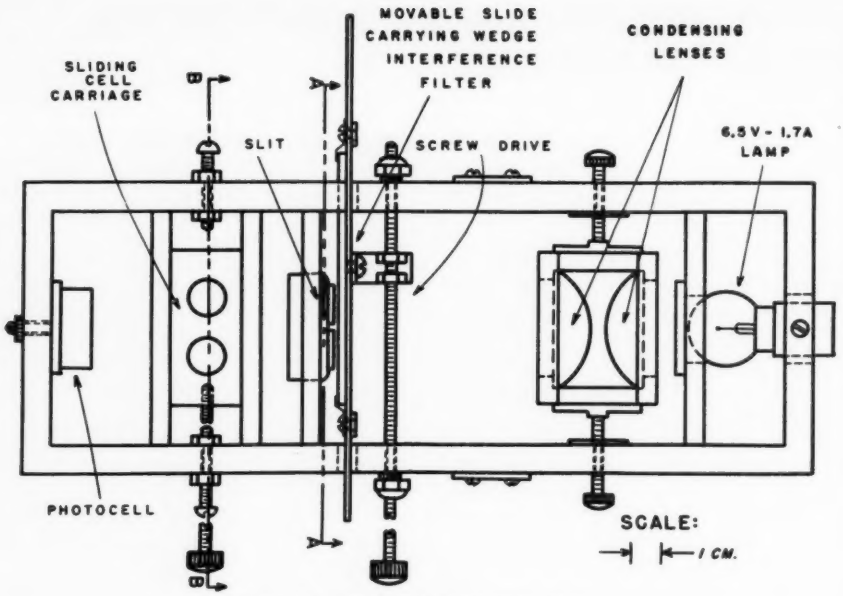


Figure 2. Top View of Spectrophotometer

each portion of the filter isolates a different spectral band and a linearly dispersed spectrum is produced. (The development of wedge interference filters has been reported by the Swiss firm of Geraetebau-Anstalt Balzers (*2*). However, these filters are not yet in commercial production (*3*).)

A few of the properties of a typical wedge interference filter are given in Table 1. Although the manufacturer states that the spectral range covered is from 400 to 700 mμ, it is a second-order spectrum and

TABLE 1
B. & L. Wedge Interference Filter

Filter dimensions	3 inch × 1 inch × 1/8 inch
Spectrum range	400 to 700 mμ
Spectrum order	2nd +
Average dispersion	5.5 mμ per mm.
Peak transmittance	25 to 35%
Spectral band pass for 1 mm. slit	10 mμ (approx.)

there is overlapping with third-order colors above 600 mμ. Interference from these higher-order wave lengths may be reduced by employing conventional cut-off filters in conjunction with the wedge filter (*2*, *4*). Experimentally, with the first wedge filter tried in our spectrophotometer we found very nearly the same dispersion noted in Table 1. Other wedge interference filters examined by the authors had slightly different average dispersion values. According to a recent announcement (*5*) the $1800 Weichselbaum-Varney flame photometer has as its monochromator one of these Fabry-Perot-type interference filters.

In the authors' analytical instrumentation course the students build "breadboard" assemblies of a polarograph, an electron-ray tube titrimeter, and so on. Accordingly, we wish to emphasize that our primary aim here was to devise a simple assemblage of easily available low-cost items—to furnish a simple, practical spectrophotometer for instructional purposes. The result is not necessarily the best arrangement for research or industrial use. For these reasons also, no attempt is made to give detailed dimensions except to indicate the scale of the drawings. Much can be left to the ingenuity of the builder—much will depend on the component parts he has available or wishes to use.

As seen in a top view in Figure 2, the framework of the spectrophotometer is a wooden box. The light source is an automobile-type lamp, actually from a microscope illuminator. Recently we have used a higher wattage bulb (G-E No. 1493, 6.5 v., 2.75 amp.) since the interference filter is quite dense optically at the violet end. The user will have to decide whether he needs a storage battery or constant-voltage transformer input. In our case, because of small load variations in our power lines, an ordinary step-down transformer of the doorbell variety often proved adequate for preliminary runs.

Two plano-convex lenses from old flashlights serve as the condensing lens system to bring the light to a focus approximately in the plane of the slit. Similar lenses are available commercially at low cost (*6*). One may note the absence of a collimating lens system to render the light rays approximately parallel before passage through the filter. If this were included, in most instances the light flux passing the filter would be too low to operate a barrier-layer or photovoltaic cell satisfactorily, so that a photoemission tube and amplifier would be required. Further, no great error seems to be introduced through the absence of a collimator. Although strongly convergent beams should not be used, it has been estimated that unless angles of incidence of light impinging on the filter are greater than 20° from the normal, the shift in wave length will not be more than 2 to 3 mμ for such inclined beams (*7*).

The metal slide bearing the filter is driven back and forth by the screw arrangement shown. Just behind the filter and almost touching it is a slit which is constructed from two parallel metal strips mounted on the face of a plastic bottle cap through which a hole is drilled. The two metal strips are adjustable to give a number of possible fixed slit widths. Finally, behind the cell carriage on the rear wall of the box is mounted

a radiant-energy receptor which in this sketch is a barrier-layer photocell (*8*).

A sectional view through *A-A* of Figure 2 is given in Figure 3 to show the mounting of the wedge interference filter. A rectangular slot is cut in the face of a metal plate and the filter is simply held against it with spring clips. A pointer is soldered to the metal slide so as to move with it, and along a millimeter scale mounted on the top of the light-tight enclosing box. The entire interior of the box, together with the various mountings, is painted flat black to minimize stray light reflections. The sectional view on *B-B* (Figure 3) shows the wooden cell carriage, drilled to hold 1-cm. cylindrical cuvettes, and movable so as to interpose first one and then the other cell in the light beam.

The accompanying photograph helps to visualize the assembly. An idea of over-all size can be gained by noting the 100-mm. scale on the top section of the box. The two variable resistors at the lower left are galvanometer shunts.

For the most accurate wave-length calibration of the millimeter scale a primary standard such as the spectral lines from a mercury arc probably should be used.

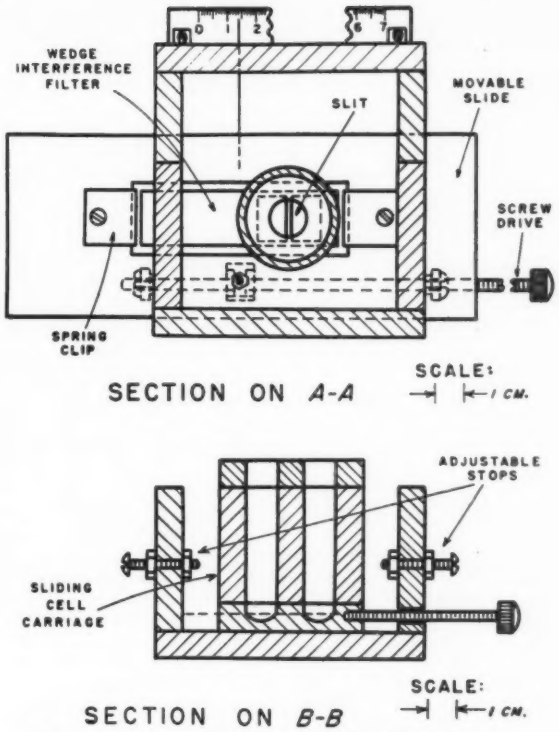


Figure 3. Sectional Views Through A-A and B-B of Figure 2

TABLE 2
Wave-Length Calibration and Dispersion Data

Scale reading, mm.	*Wave length of absorption maxima, mμ*	*Dispersion, mμ/mm.*
55.0	441	
		5.75
61.0	475	
		5.60
70.5	529	
		5.63
80.5	585	
	Weighted average	5.64

Too, the Bureau of Standards considers the use of a didymium glass reference inferior from the standpoint of time, convenience, and so on, to the direct use of line sources (*9*). Nevertheless, lacking a primary standard of this sort, a didymium glass filter is an excellent and readily available secondary wave-length standard. The Corning No. 5120 glass is the most suitable for this purpose. This glass contains the rare earth ions praseodymium and neodymium, both of which exhibit strong and narrow absorption bands in the visible spectrum. The calibration procedure is quite simple. The exit slit is set to a fairly narrow opening, say 1 mm., and the transmittance of the filter is measured against an air standard at wave lengths in the vicinity of the known absorption maxima and minima. Typical dispersion data are recorded in Table 2. The scale readings in the first column are those at which absorption maxima were observed. The wave lengths of these maxima in the second column are those reported by the Bureau of Standards (*9*). From the average dispersion (a weighted value, taking into account the linear distance between absorption maxima) a table was constructed relating scale readings in millimeters to wave lengths in millimicrons. Thus far, using these calibration data, the wave-length readings on various substances have checked previously determined

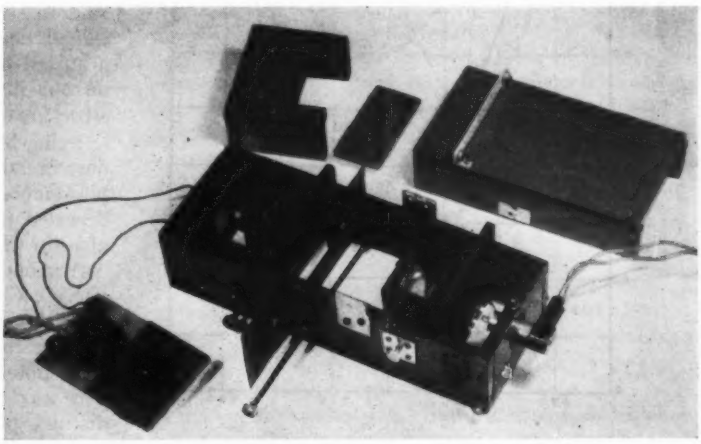


Figure 4. Complete Spectrophotometer Showing Arrangement of Light Source, Lens System, Interference Filter, Cell Carriage, Photocell, and Top Sections Bearing the Millimeter Scale

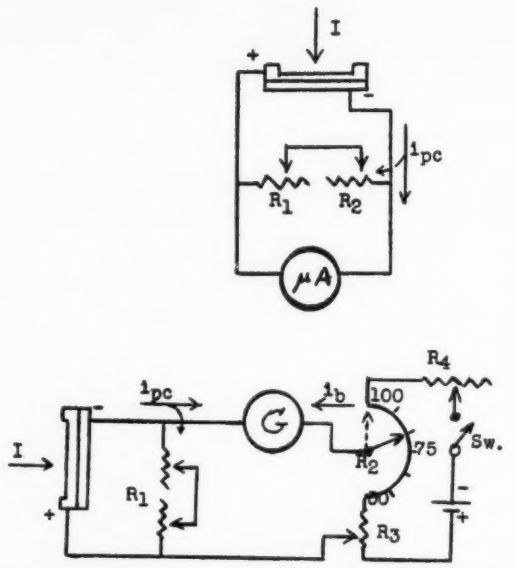


Figure 5. (*a*) *Top:* Direct Measurement of Photocurrent with Shunted Variable Resistors to Control Current Flowing Through the Meter (*b*) *Bottom:* Potentiometric Measurement of Photocurrent

values to better than 2 mμ. Newer wedge filters examined have slightly higher average dispersion values but the linearity of dispersion, in general, is as good as

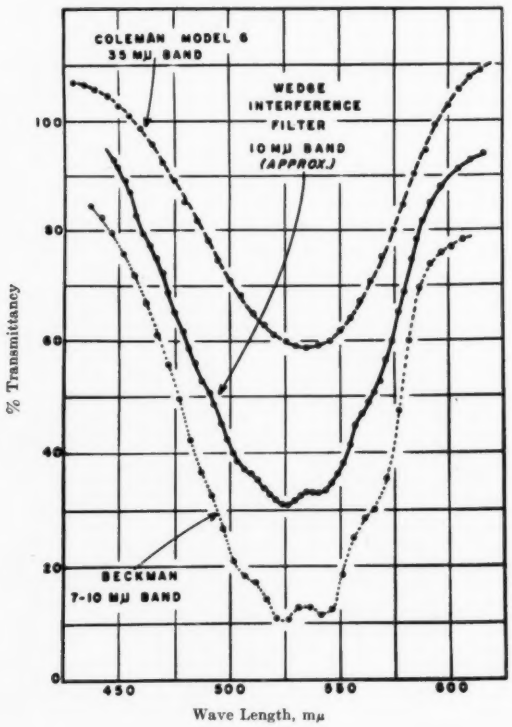


Figure 6. Transmittancy-Wave-Length Characteristics of a Permanganate Ion Solution, 0.02 mg. Mn/ml. Data Taken from Three Different Spectrophotometers

that shown in Table 2. Of course, each wedge must be individually calibrated by the user.

Most of the measurements described were made with a barrier-layer photocell connected directly to a shunted Rubicon spotlight galvanometer whose sensitivity was 0.008 μamp./mm. Two variable resistors, of 5000 and 500 ohms resistance, were used for adjusting the galvanometer to 100 per cent transmittance with the reference substance in the light beam (Figure 5*a*). It is recognized that not all laboratories will have such a sensitive galvanometer available exclusively for this use. Thus a circuit of the sort shown in Figure 5*b* will be helpful since it requires only standard components. Resistance R_1 corresponds to the meter and shunt of Figure 5*a*. By means of a simple pointer galvanometer the voltage drop across R_1 is balanced against that across R_2-R_3 at the right. The whole righthand portion of the circuit will be recognized as the usual student potentiometer set-up found in all physical chemistry and physics laboratories. Transmittancy readings are taken directly from the slidewire scale. For other circuits, including those in which photoemission tubes with a simple amplifier are used, the reader is referred to standard works on the subject (*10*). Multiplier phototubes were not considered here because they require a rather complex voltage supply.

Although the calibration data obtained with the didymium filter best show the high resolving power of the wedge-slit combination, the transmittancy-wave length curve from which the data were obtained is not reproduced here because its appearance is familiar to many. As one graphical example of results, however, consider Figure 6. Here are plotted the transmittancy-wave-length characteristics of a permanganate ion solution as determined with three spectrophotometers. (The transmittancy values are only relative since the curves have been deliberately spaced as an aid to comparison.) The curve at the top was obtained with a diffraction grating Coleman Model 6 spectrophotometer whose spectral band pass is fixed at 35 mμ. Only a smooth curve is the result. The lowest curve was obtained using a Beckman Model DU instrument, so adjusted that its band width would approximate that on our instrument. It shows the characteristic fine structure of the permanganate ion absorption spectrum.

Lying between these two curves is one plotted from data read on our wedge-interference-filter spectrophotometer. That the resolution is high is clearly revealed from the fine structure it too shows. Note also the close coincidence with wave-length values for corresponding maxima and minima in the Beckman curve. (The larger number of plotted points here arises from the intervals at which the millimeter scale may be most easily read.)

As noted very early in this discussion, the primary aim was to devise a simple apparatus having some distinct pedagogical values. For those who are interested, we are sure many apparatus refinements can be made to produce an instrument that will be more than satisfactory for routine industrial or research use.

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RECLAIMING SILVER FROM SILVER CHLORIDE RESIDUES

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IN MANY universities budget considerations make it desirable to have available a simple and economical method for the reclaiming of silver from silver chloride precipitates in the quantitative analysis course. In some cases three or four analyses requiring the use of silver nitrate are run by each student, and often the cost of this reagent alone exceeds the income provided by student fees. A convenient method for accomplishing the conversion of silver chloride to silver is proposed which takes advantage of the fact that silver oxide is less soluble in concentrated base than is silver chloride.

The solubility product constants for silver chloride and silver hydroxide are 1.0×10^{-10} and 1.0×10^{-8}, respectively. From this it follows that the concentration of silver ion in equilibrium with a precipitate of silver oxide in a solution five molar in hydroxyl ion is 2.0×10^{-9} mols/liter, while the concentration furnished by a precipitate of silver chloride in the same solution is 1.0×10^{-5} mols/liter. Hence under these conditions silver chloride furnishes 5000 times the silver ion concentration required for precipitation of silver oxide, and this reaction can be used to convert silver chloride to silver oxide.

$$AgCl + OH^- \rightarrow AgOH + Cl^-$$
$$2\,AgOH \rightarrow Ag_2O + H_2O \quad \text{(spontaneous)}$$

Student precipitates collected in the laboratory are filtered, washed with dilute nitric acid, and transferred to a large beaker where they are covered with about three times their apparent volume of five-molar sodium hydroxide. The mixture is boiled for 15 minutes with stirring to break up clumps of silver chloride, filtered, and washed with tenth-molar sodium hydroxide. The resulting residue is treated again as above and then transferred to a crucible or casserole and ignited at 500°C. for 15 minutes, converting the silver oxide to metallic silver. After cooling, the spongy solid is washed several times in the crucible with concentrated ammonium hydroxide to remove traces of silver chloride which were not converted, and then heated until the silver melts. A large Büchner funnel is convenient for the filtrations and a muffle furnace is desirable but not necessary for the ignitions.

The purity of silver obtained in this manner is primarily dependent upon the purity of the silver chloride used as starting material. In one case where specially prepared silver chloride was used, the silver obtained was 99.2 per cent pure. In most cases silver obtained from student precipitates is in excess of 96 per cent pure, the principal impurities being silver chloride and sodium hydroxide. By dissolving in nitric acid, filtering to remove traces of silver chloride, and diluting, an acceptable reagent is had for the determination of chloride gravimetrically or, if standardized, volumetrically.

This method may also be used with equally satisfactory results on the chromate and thiocyanate of silver or on mixtures of the chloride, chromate, and thiocyanate.

Due to the explosive nature of silver ammino compounds, homogeneous precipitation from ammoniacal solution is not practical.

It should be pointed out that other methods of effecting this conversion exist. Among those used are electrolysis, fusions with sodium hydroxide and sodium carbonate, and procedures involving organic reductants.

GERHART JANDER

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THE relative freedom from distractions in a small and somewhat isolated university can be reflected in the volume and excellence of the research and literary output of the faculty. A good case in point is the record of Gerhart Jander, who for 16 years headed the chemistry department at Greifswald, a small Pomeranian city in Northern Prussia.

He was born on October 26, 1892 in Alt-Döbern (Lausitz) where his father was headmaster in the teacher's training school. His university work in natural science and chemistry was taken in Munich and then Berlin, where his doctorate was conferred in 1917. His dissertation was based on a laboratory study of telluric acids. Dr. Jander served as assistant, Privatdozent, and extraordinary professor at Berlin and Göttingen and then was attached to the Kaiser Wilhelm Institut für physikalische Chemie in Berlin-Dahlem. Between 1935 and 1951 he was director of the Chemisches Institut of the University at Griefwald, and in 1951 he became director of the Anorganisch-Chemisches Institut of the Technical University at Berlin-Charlottenburg.

His research activities have been many and varied. Through the influence of his doctorate mentor, A. Rosenheim, the years immediately following his attainment of this degree were devoted to the study of iso- and heteropoly acids and isopoly bases. By employing a variety of chemical and physicochemical procedures, such as diffusion measurements, conductometric, potentiometric, and thermometric titrations, determination of light absorption, along with preparative and analytical studies, he, assisted by numerous collaborators, opened up entirely new viewpoints in this complicated field of research. By virtue of his fundamental explorations, much light was shed particularly on the molecular size of the individual representatives of this class of materials in aqueous solution and on their genetic relationships to each other.

Closely connected with the foregoing were the experimental studies of diffusion and dialysis as means for investigating the state of dissolved materials. The investigation of these methods dealt, for example, with the basic problem as to the conditions under which the diffusion coefficients of dissolved substances, molecules as well as ions, can be called upon with regard to molecular weight determination. Studies along this line are still being made in Jander's laboratory.

In another direction, considerable attention has been given to problems of analytical chemistry. In particular, conductometric titrimetric procedures have been developed. The design of special apparatus and sensitive instruments has enabled him to extend the application of this method to the determination of smaller and smaller amounts of materials in the presence of a relatively large quantity of foreign electrolytes which ordinarily interfere, and of electrolytes in "nonaqueous" solvents, and to cases that hitherto had barely been considered. Without the development and mastery of conductometric measurement procedures, it would hardly have been possible to have carried out the studies in nonaqueous or water-similar solvents, a field of research in which Professor Jander has specialized for many years.

Almost without interruption since about 1935, he and his advanced students have been dealing with the chemical and physicochemical behavior of materials when dissolved in nonaqueous but water-similar solvents. These liquids, like pure water, hardly conduct any electric current, whereas their solutions of many inorganic and organic compounds are fairly conductive. Jander has compared the chemistry of such water-similar solvents (*e. g.*, H_2S, HCN, HNO_3, SO_2, $(CH_3CO)_2O$, I_2, etc.) with each other and with water. Much information of great theoretical importance has resulted, and likewise usable procedures for preparative chemistry. The most remarkable findings have led him recently to extend his studies to the behavior of materials dissolved in fused salts such as $HgBr_2$, $SbBr_3$, $AlBr_3$, *i. e.*, to melts of salts which conduct but slightly when pure. Surprising analogies have been found here also.

The results of the extensive and multifarious researches carried out by him and under his guidance have been published in the well-known German journals. In addition, Professor Jander has issued accounts of certain areas of his studies in book form. The following compilation contains also the textbooks and laboratory manuals which he has composed for the use of chemistry students:

"Kurzes Lehrbuch der anorganischen Chemie," G. JANDER AND H. SPANDAU, 5th ed., Berlin, 1952.

"Lehrbuch der analytischen und präparativen analytischen Chemie," G. JANDER AND H. WENDT, Leipzig and Stuttgart, 1952.

"Einführung in das anorganisch-chemische Praktikum (einschl. der quantitativen Analyse)," G. JANDER AND H. WENDT, 2nd ed., Leipzig, 1951.

"Massanalyse. Theorie und Praxis der klassischen und elektrochemischen Titrierverfahren," G. JANDER AND K. F. JAHR, 6th ed., Berlin, 1952.

"Membranfilter, Cella- und Ultrafeinfilter," G. JANDER AND J. ZAKOWSKI, Leipzig, 1929.

"Schwebstoffe in Gasen (Aerosole)," A. WINKEL AND G. JANDER, Stuttgart, 1934.

"Die konduktometrische Massanalyse und andere Anwendungen der Leitfähigkeitsmessung auf chemische Probleme," G. JANDER AND O. PFUNDT, 3rd ed., Stuttgart, 1945.
"Handbuch der analytischen Chemie" (in about 30 volumes with collaboration of numerous eminent colleagues), edited by R. FRESENIUS AND G. JANDER. Published since 1942. Ten partial volumes with a total of about 4000 pages have appeared thus far.
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THE SEPARATION OF CALCIUM AND STRONTIUM IN SEMIMICRO QUALITATIVE ANALYSIS

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THE cations of Group IV in qualitative analysis include the barium, strontium, and calcium ions. In all procedures barium is separated first by precipitation as barium chromate in an acetic acid solution. No difficulty is encountered usually in this step. The following step involving the separation of strontium and calcium is the troublesome one.

Current methods for the separation of strontium from calcium usually give mediocre results in the hands of the beginning student. These methods include precipitation as chromate in alcohol-water solution, precipitation as sulfate and precipitation as ferrocyanide. Methods using anhydrous solvents, as butyl cellosolve or acetone, to dissolve calcium nitrate are likewise unsatisfactory in the hands of students.

A method which gives an excellent separation of calcium and strontium was devised by Willard and Goodspeed.[1] This involves the precipitation of strontium nitrate in 80 per cent nitric acid solution. By this method the above authors were able to separate quantitatively strontium from calcium. The only drawback encountered in this method is the use of 100 per cent (fuming) nitric acid which is somewhat hazardous. This difficulty can be overcome by the use of ordinary 72 per cent (concentrated) nitric acid. The method is no longer quantitative with acid of this concentration, but an excellent qualitative separation is achieved. The loss of strontium amounts only to a few tenths of a milligram which is insignificant in qualitative analysis. The following procedure is suggested:

A. Precipitation of Group IV and the Separation of Barium. This is carried out in the usual manner: $BaCO_3$, $SrCO_3$, and $CaCO_3$ are precipitated by the addition of ammonium carbonate. This precipitate is dissolved in acetic acid and barium chromate precipitated by the addition of potassium chromate.

B. Reprecipitation of $CaCO_3$ and $SrCO_3$. The solution from the separation of barium is rendered basic by the addition of ammonium hydroxide. Ammonium carbonate is added, the solution is warmed and centrifuged. The supernatant liquid is discarded and the precipitate is washed two times with hot water. These washings are discarded.

C. Separation of Calcium and Strontium. The precipitate of $CaCO_3$ and $SrCO_3$ (free of liquid) in the tube is dissolved by the cautious, dropwise addition of 72 per cent (concentrated) nitric acid. After effervescence has ceased, an additional 5 ml. of concentrated nitric acid is added. The mixture is stirred vigorously for a minute or two, then the tube is placed in a beaker containing cold water and allowed to stand for 10 minutes with occasional stirring. (Caution, do not allow any water to get into the tube. This will dilute the acid and render the $Sr(NO_3)_2$ precipitate soluble.) The mixture is centrifuged and the supernatant liquid decanted into a beaker containing 20 ml. of water. This solution is saved for the calcium test. The precipitate is stirred up with 1 ml. of 72 per cent nitric acid, the mixture centrifuged, and the liquid discarded.

D. Identification of Strontium. The precipitate, $Sr(NO_3)_2$, is dissolved in 3 ml. of hot water. A flame test is made on this solution (Sr = crimson). If barium were incompletely separated it would also be precipitated as an insoluble nitrate. Five drops of 1 *M* ammonium sulfate is added to the solution and the mixture heated to boiling. The formation of a white flocculent precipitate of $SrSO_4$ confirms the presence of strontium.

E. Identification of Calcium. Concentrated (15 *M*) ammonium hydroxide is added cautiously to the *dilute* nitric acid solution from the stontium separation until the solution is basic (litmus test). Two ml. of saturated (0.25 *M*) ammonium oxalate are added to this basic solution. A finely divided white precipitate of CaC_2O_4 indicates the presence of calcium.

The above procedure has been used in the author's classes during the past year. The accuracy has been above 95 per cent with this new procedure compared with accuracies below 70 per cent with other procedures. As little as 1 mg. of Sr can be detected in the presence of 50 mg. of Ca and vice versa, using this procedure. The students were reminded about the corrosive nature of concentrated nitric acid, hence no accidents have arisen in using this reagent.

[1] WILLARD, H. H., AND E. W. GOODSPEED, *Anal. Chem.*, 8, 414 (1936).

PERMANGANATE STANDARDIZATION WITH A BECKMAN SPECTROPHOTOMETER

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STUDENTS of quantitative analysis often lose valuable time in standardizing several portions of 0.1 N $KMnO_4$ solution. The following spectrophotometric method is suggested as a time-saver that might be employed by the instructor when a Beckman model B Spectrophotometer is available. Besides saving time, this method affords the student an opportunity, under the direction of his instructor, of gaining a nodding acquaintance, at least, with a useful spectrophotometric method. Also, the instructor may employ it as an introduction to a discussion of this method as applied to other[1] quantitative determinations.

PROCEDURE

The transmittancy of a $KMnO_4$ solution of *approximately* 0.1 N is compared by means of a calibration curve with the transmittancy of a $KMnO_4$ solution of known strength. The solution may then be used, employing the factor obtained from the calibration curve, or may be adjusted to the exact normality by suitable dilution.

Preparation and Standardization[2] *of Solution.* Weigh on a watch glass approximately 3.2 g. of reagent-grade $KMnO_4$ crystals. Dissolve in 1.0 l. of distilled water, and heat in a covered beaker for 2 hours on a steam bath. Allow the covered beaker to stand overnight, filter through a sintered-glass filter, and store in a dark, glass-stoppered bottle. Formation of a brown crust on the inner surfaces of the bottle indicates a change in the composition of the solution.

Dissolve 20 g. of reagent-grade NaOH pellets in 80 ml. of water and cool to room temperature. Weigh, to the closest 0.1 mg., three 0.2-g. portions of dried arsenious oxide of known purity into 250-ml. wide-mouth Erlenmeyer flasks. Add 10 ml. of the cooled NaOH solution, and allow to stand 8–10 minutes with occasional swirling, or until the sample is dissolved. Dilute to 100 ml. and add 10 ml. concentrated HCl and 1 drop 0.002 M potassium iodide or iodate solution. Titrate with $KMnO_4$ solution until a faint pink color persists for 30 seconds or more. Run a blank determination. From the net volume of permanganate, corrected for buret calibrations and the blank, and from the weight and purity of the standard compute the normality of the permanganate. The average deviation should not exceed one part per thousand.

Calibration of Standards.[3] In a series of three 500-ml. volumetric flasks place, respectively, 2.0, 2.5, and 3.0 ml. of the standardized 0.1 N $KMnO_4$ solution; dilute to volume with distilled water and mix thoroughly. The normalities of these standards are approximately $N/2000$ times the factors 0.8, 1.0, and 1.2. Prepare just before use and proceed immediately as follows.

Transfer a sufficient portion of each calibration standard to one of a pair of matched (preferably quartz) cells. Set the distilled water in the other cell at 100 per cent transmission on 530 mμ and record the transmittancy of the calibration standards. The transmittancy of the solution with the factor 1.2 should be *approximately* 49 per cent. Plot the observed values on semilog graph paper using the factors 0.8, 1.0, and 1.2 as ordinates so that each division unit represents a factor change of 0.01. The points fall on a straight line.

Routine Tests. Potassium permanganate stock solution, approximately 0.1 N, should be prepared in the same manner as in the calibration procedure. Dilute 2.5 ml. of this stock permanganate solution to 500 ml. with distilled water. Place a sufficient quantity of this diluted solution in one of the matched cells. Measure the transmittancy of this sample against distilled water set at 100 per cent transmission on a wave length of 530 mμ, and obtain the factor from the prepared calibration curve. Multiply the normality of the standard solution by this factor to obtain the normality of the stock solution.

$$N \text{ of stock solution} = \text{factor} \times N \text{ of standard solution}$$

REMARKS

The temperature of the distilled water and solutions must be the same during all transmission measurements.

The stock solution (if factor is greater than 1.0) may be diluted to the proper normality (0.1 N) by application of the following formula

$$\frac{1000}{\text{factor}} = \text{ml. of stock solution to be diluted to 1000 ml.}$$

It is preferable, however, to use the permanganate solution without further dilution, and to correct results by means of the factor.

Better results seem to be obtained if the $KMnO_4$ solutions are filtered with sintered-glass filters. Such filters may be cleansed of MnO_2 residue with the usual dichromate solution.

[1] LINGANE, J. J., AND J. W. COLLAT, *Anal. Chem.*, **22**, 166 (1950).

[2] PIERCE, W. C., and E. L. HAENISCH, "Quantitative Analysis," John Wiley & Sons, New York, **1948**, pp. 214, 216.

[3] FISTER, H. J., "Manual of Standardized Procedures for Spectrophotometric Chemistry," Standard Supply Corp., New York, **1950**, method P-45.1.

COULOMB'S LAW AND THE QUANTITATIVE INTERPRETATION OF REACTION RATES, II[1]

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As in the former paper of this series (*1*), the following discussion is pertinent only when electrostatic influences upon reactants in solution predominate over specific effects such as those arising from the solvent due to solvation and other causes (*2*).

Papers dealing with Coulomb's law and both the qualitative and quantitative interpretation of ion-ion reaction rates have already been published (*1*, *2*). This paper deals with Coulomb's law and the quantitative interpretation of reaction rates between ions and dipolar molecules and between dipolar molecules and dioplar molecules. The equations are derived from Coulombic energy considerations coupled with the Arrhenius equation. This method of approach has the disadvantage of applying only to dielectric constant effects but has the advantages of simplicity and of application to various electrically charged and electrically unsymmetrical combinations of reactants. The salt effect is still best approached, perhaps, through complex formation and activity coefficients.

We shall first consider reactions between ions and dioplar molecules.

The mutual potential (potential energy), E_c, between an ion and a dipole is

$$E_c = \frac{z\,\epsilon\,\mu\cos\theta}{Dr^2} \tag{1}$$

In this equation higher powers than the first of the distance of separation of centers of charge in the dipole are neglected. Amis (*3*) has derived the Coulombic energy for this type of reactant pair to be

$$\Delta E_c = \frac{z\,\epsilon\,\mu\cos\theta}{D_1D_2r^2}\,\Delta D \tag{2}$$

which can be written

$$\Delta E_c = \frac{-z\,\epsilon\,\mu\cos\theta}{r^2}\left(\frac{1}{D_2}-\frac{1}{D_1}\right) \tag{3}$$

and for the case of head-on alignment we have

$$\Delta E_c = \frac{-z\,\epsilon\,\mu}{r^2}\left(\frac{1}{D_2}-\frac{1}{D_1}\right) \tag{4}$$

Following a procedure similar to that used in deriving equation (14), reference (*1*), we obtain

$$\ln k'_{D_2} = \ln k'_{D_1} + \frac{z\,\epsilon\,\mu}{kTr^2}\left(\frac{1}{D_2}-\frac{1}{D_1}\right) \tag{5}$$

and

$$\ln k'_{D\,-\,D} = \ln k'_{D\,-\,\infty} + \frac{z\,\epsilon\,\mu}{DkTr^2} \tag{6}$$

In equations (1) to (6) $z\,\epsilon$ is the charge in the ion and μ is the dipole moment of the molecule. According to equation (6) if D is increased the rate should decrease for a positive ion and vice versa for a negative ion. If D is decreased the rate increases for positive ions and vice versa for negative ions. These predictions are in harmony with those of the Amis-Jaffé equation (*4*).[2] The specific velocity constants are preferably those at zero ionic strength. Probably the better value of the moment to use for zero ionic strength in equation (6) would be μ_0^* which is related to μ by the Onsager relationship

$$\mu_0^* = \frac{\mu(n^2+2)D}{2D+n^2} \tag{7}$$

and equation (6) then becomes

$$\ln k'_{\substack{D\,-\,D\\ \kappa\,=\,0}} = \ln k'_{D\,-\,\infty} + \frac{z\,\epsilon\,\mu_0^*(2D+n^2)}{D^2kTr^2(n^2+2)} \tag{8}$$

The symbols μ_0^* and μ^* are the external characteristic moments of a molecule when immersed in a dielectric medium and when the ionic strength is zero and not zero, respectively, and n is the internal refractive index.

This equation contains parameters μ_0^*, r, D, and n and assumptions would have to be made concerning them since their values are ordinarily not found in the literature. One could assume reasonable values of r and n and plot

$$\log k'_{\substack{D\,-\,D\\ \kappa\,=\,0}} \quad \text{versus} \quad \frac{z\,\epsilon\,(2D+n^2)}{D^2kTr^2(n^2+2)}$$

and if a straight line with a slope which gives a reasonable value of μ_0^* is obtained then theory is coinciding with experiment.

A simple check might be made using equation (6) by assuming or taking from the literature a value of μ and plotting $\ln k'_{D\,-\,D}$ versus $1/D$ and if a straight line is obtained, the slope of which gives a reasonable value of r, then theory and experiment are in agreement.

The D values used in these data would as a first approximation be the macroscopic dielectric constants of a mixed solvent pair of various controlled compositions so chosen as to give the desired dielectric constants.

A test of equation (6) was made using the data of Amis and Holmes (*5*) on the inversion of sucrose by hydronium ion at 41°C. Amis and Jaffé (*4*) used these

[1] This work was done on a contract made by the Institute of Science and Technology of the University of Arkansas with the Office of Naval Research.

[2] See also ref. (*3*), pp. 159–74.

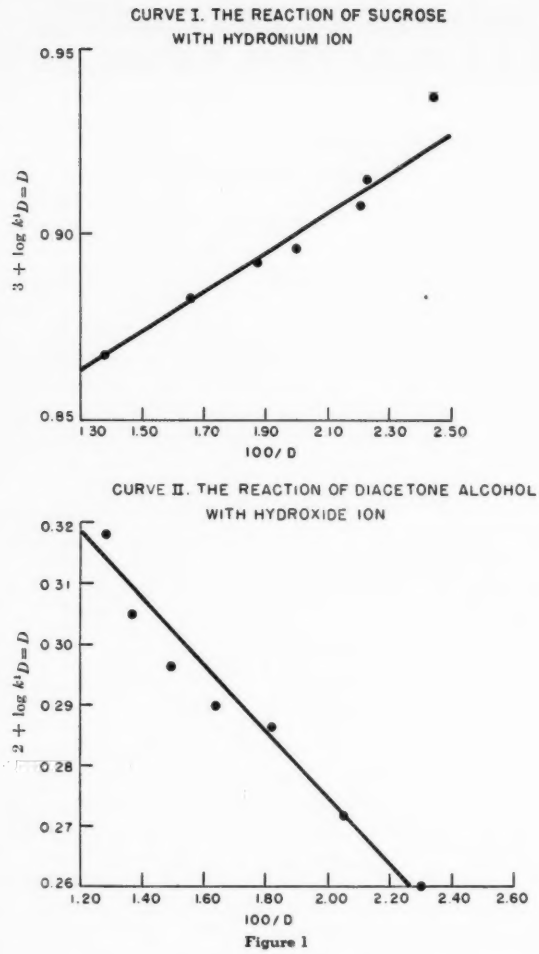

Figure 1

authors' data at 21°, 31°, and 41°C. The straight line given in Figure 1, when values of log $k'_{D=D}$ are plotted as ordinates against values of $1/D$ as abscissae has the right direction of slope as predicted by equation (6). The value of this slope using Landt's (*6*) datum of 3.4 Debye units for the moment, μ, of sucrose gives a value of r of 5.5×10^{-8} cm. for the distance of approach, r, for hydronium ion and sucrose to react. This is a reasonable value of r and compares favorably with the value of 3.5×10^{-8} cm. found by Amis and Jaffé (*4*) for this reaction using their kinetic equation.

Using Akerlöf's data for the reaction of the negative hydroxide ion with the diacetone alcohol molecule at 25°C. in water-ethyl alcohol solvents, one gets the log $k'_{D=D}$ versus $1/D$ plot given in Figure 1. The data are somewhat scattered, as was found by Amis, Jaffé, and Overman (*7*). Assuming a moment of $\mu = 4.0 \times 10^{-8}$ as did these authors, one finds a value of 6.0×10^{-8} cm. from the slope of the log $k'_{D=D}$ versus $1/D$ curve. Amis, Jaffé, and Overman found $r = 1.0 \times 10^{-8}$ cm. using the Amis-Jaffé equation. The r parameter for this negative ion-dipolar molecule reaction thus has a reasonable value calculated from the present theory. A lower value of the moment would give a lesser value of r. Ln $k'_{D=D}$ and not ln $k'_{\kappa=0,D=D}$ was used here but since the runs were made at constant ionic strength, the error introduced is not too significant.

The disadvantages of the present method of approach to the influence of the dielectric constant effect upon reaction rates is that it applies only to electrostatic effects arising from charges and/or moments possessed by reactants immersed in a dielectric medium. The methods depending on activity coefficients and concentrations of intermediate complex and reactants can be applied also to ion atmosphere effects.

We will now consider reactions between dipole-dipole reactants. Thus for dipole-dipole reactants the dielectric constant influence on the rate is given by the following considerations where all powers higher than the second of distances of separation of charge centers in dipoles have been neglected. The Coulombic energy of the dipolar reactants is

$$E_c = \frac{2\mu_1\mu_2 \cos\theta_1 \cos\theta_2}{Dr^3} + \frac{\mu_1\mu_2 \sin\theta_1 \sin\theta_2}{Dr^3} \qquad (9)$$

and for head-on alignment

$$E_c = \frac{2\mu_1\mu_2}{Dr^3} \qquad (10)$$

thus

$$\Delta E_c = \frac{2\mu_1\mu_2}{r^3}\left(\frac{1}{D_2} - \frac{1}{D_1}\right) \qquad (11)$$

and

$$\ln k'_{D=D} = \ln k'_{D=\infty} - \frac{2\mu_1\mu_2}{DkTr^3} \qquad (12)$$

Equation (12) would predict a linear relationship between ln $k'_{D=D}$ and $1/D$ as a first approximation so long as the moments μ_1 and μ_2 can be used as the simple moments *in vacuo* of the reactant dipoles. Further,

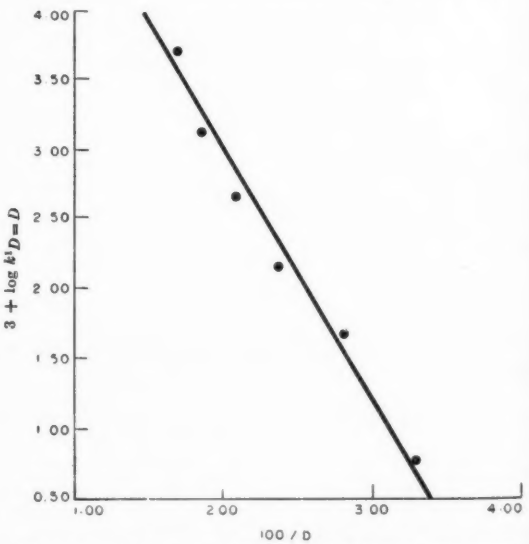

Figure 2. The Reaction of Tertiary Butyl Chloride with Water

the equation would predict an increase of ln $k'_{D=D}$ as D increases. An equation by Laidler and Eyring (*8*) gives an increase of ln $k'_{D=D}$ with increasing dielectric constant D. These authors, however, obtain from free energy and activity coefficient considerations an expression which relates ln $k'_{D=D}$ to $(D-1)/(2D+1)$ in a linear way under certain restricted conditions.

In Figure 2 a plot of ln $k'_{D=D}$ versus $1/D$ is given for the reaction between the dipolar reactants water and tertiary butyl chloride in ethyl alcohol-water solvents of various compositions at 25°C. The data are those of Hughes (*9*). The data on such a plot may be considered as a straight line, although there is a slight appearance of an extremely skewed S. The deviation from theory may be attributed to lack of precision. Taking the slope of the line and equating the slope to $-2\ \mu_1\ \mu_2/kTr^3$ as required by equation (12) one obtains a value of r of 0.75×10^{-8} cm. if the moment of water and the moment of tertiary butyl chloride are taken to be 1.84×10^{-18} and 2.10×10^{-18} (*10*), respectively.

The value of r obtained using the approximations discussed in deriving equation (10) is possibly somewhat low, but it is not off perhaps by more than a factor of 4 or 5. This is gratifying, considering the simplicity of equation (12), the ease of visualizing its predictions, and the approximations made in its derivation.

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WITH OR WITHOUT SECONDARY-SCHOOL CHEMISTRY

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Among many professors who teach general chemistry in our colleges and universities there persists the opinion that it makes little difference whether their students have or have not had previous work in secondary-school chemistry as to the probability of success or failure in college chemistry courses. To quote a few examples:

> The failure rates will be about the same if the instructor uses good judgment in the early lectures about defining new terms, working problems, balancing equations, etc.
>
> I have no numerical data in this regard, but am fairly certain from hearing of different cases, that there is little difference.
>
> No records, but we believe there to be no strong correlation between high-school records and performance in this course.
>
> Unless the secondary school was a technical high school, we find that those students without chemistry do better. The reason may be that the student's efforts are less, feeling that this is repetition. When the students realize it is not, then it is too late—a little knowledge is dangerous.

Is this belief correct? "No!!! No!!! A thousand times no!!!"

During the past year a questionnaire was sent from the Naval Academy to the heads of departments of chemistry in representative colleges and universities throughout the country asking data with regard to courses in general chemistry. One of the questions asked was as to whether there had been observed any difference in failure rates between those who had had secondary-school chemistry and those who had not had previous chemistry. This article is written as the result of the answers, quantitative and qualitative, received from the questionnaire.

In every case where quantitative data are available it was shown that *the rate of failures among those who*

have not presented chemistry for entrance is much greater than for those who have had secondary-school chemistry.

FAILURE RATES

From 1941 until the class which entered in 1947, the Naval Academy required high-school chemistry for entrance on certificate or by examination. Since the class which entered in 1948, 14.0 per cent of the entering midshipmen have had no chemistry previous to entrance. From this group there has been a failure rate of 15.4 per cent for the first term, which is three times the average rate of failure for the whole class.

TABLE 1
1948–53 (Five Years), First Term, General Chemistry

Total entering midshipmen	5338	
Entering without chemistry	756	14.0%
Entering with high-school chemistry	2988	56.1%
Entering with college chemistry	1594	29.9%
Withdrawals before examination	170	3.2%
Term failures, without chemistry	109	15.4%
Term failures, with high-school chemistry	139	4.8%
Term failures, with college chemistry	24	1.6%
Term failures, total	272	5.3%

The data in Table 1 show that although the overall failure rate for the first term has been only 5.3 per cent we can expect a failure rate among those who have not had previous chemistry of 15.4 per cent, for those with only high-school chemistry of 4.8 per cent, and for those who have had college chemistry of only 1.6 per cent. It is also worthy of note that the withdrawals before examination for those without previous chemistry are 2.5 times as great as for those entering with chemistry. The statistics therefore definitely show the advantage of having had previous chemistry.

As this difference in failure rates consistently appeared every year in our analysis of failures, we inquired in the questionnaire as to the difference in failures in the colleges and universities for those with and without high-school chemistry. The returns from the colleges roughly paralleled those of the Naval Academy.

On the average, the failure rate for those without entering chemistry in the civilian colleges was 20.5 per cent, and for those with high-school chemistry was 7.6 per cent. In two schools, where separate courses in general chemistry were operated for those with and without chemistry, the failure rate was 14.3 per cent for the courses which required entering chemistry, and 25.9 per cent for those which did not.

In requesting the data on variation of failure rates for those with and without entering chemistry, it was not asked whether those with and without chemistry were allowed to take the same course. However, in the answers received which represented 86 courses, it was indicated that in 45 courses students were present both with and without entering chemistry, in 17 courses only those entering with chemistry took the course, and in nine cases the course was only for those without high-school chemistry.

In several colleges there were two separate courses in beginning or general chemistry, one for those with and one for those without high-school chemistry. This seems to be a most logical method of solving the problem, as one professor wrote:

> Note that those who have not had chemistry before take our Chemistry 1 and 2. Those with previous chemistry take course 3 and 4. We are quite confident that this is a wise arrangement; for we have tried both systems.

GRADES VERSUS PREVIOUS PREPARATION

In the Naval Academy all entering plebes have to take the same course in general chemistry, no matter what their background, no matter whether they have been interested in science in high school or not. The statistics show that a good student will pass the course even if he has not had a previous one, for if 15.4 per cent have failed then 84.6 per cent of this group have passed. There are many failures among those who have had secondary-school chemistry, and even some failures among those who have had equivalent college chemistry. Most of these failures are because of lack of application, an inability to study, the stiff pace that is set, a tendency to "ride the velvet" and find oneself unprepared when the examination arrives.

Analysis of failure represents merely the analysis of an end point. It then becomes interesting to see whether the data shows the same correlation at the top of the grade scale as at the bottom. Have the better men all had previous chemistry? What percentage of those without high-school chemistry are in the top 50 per cent of the class? What is the probability that a man without high-school chemistry will get an "A" in the course?

At one of the larger universities a correlation curve was set up between grades and those who had and had not had secondary school chemistry, both of these groups taking the same course. The data obtained are shown in Table 2.

TABLE 2

Grade	*With HS chem., %*	*Without HS chem., %*	*Ratio*
A	9.9	3.8	2.6:1
B	23.1	16.3	1.4:1
C	42.9	37.5	1.15:1
D	21.1	30.0	1:1.4
F	3.1	12.5	1:4

Note first that the failure rate is four times as great for those who have not had high-school chemistry. If A counts as 4 credits, B as 3, C as 2, D as 1, and F as zero, then the average mark for those having high-school chemistry is 2.16 or C+, and for those without high-school chemistry was 1.84 or C−, a difference of 16 per cent.

A study was made of the grades in the first term, 1951–52, in the general course in chemistry at the Naval Academy. This included 149 men who had not had any previous chemistry, 564 who had had high-school chemistry only, and 306 who had had some col-

lege chemistry. The results on a percentile basis are shown in Table 3 and the accompanying graph. They definitely show the superiority of those who have had

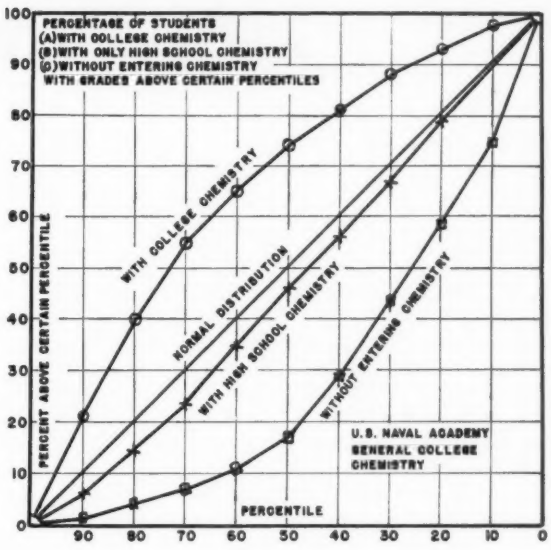
TABLE 3

	With college chemistry		*With only high-school chemistry*		*Without chemistry*	
Percentile	*Per cent in 10% bracket*	*Cumulative*	*Per cent in 10% bracket*	*Cumulative*	*Per cent in 10% bracket*	*Cumulative*
90	22.2	22.2	5.7	5.7	1.3	1.3
80	16.4	38.6	8.7	14.4	2.1	3.4
70	16.1	54.7	8.6	23.0	2.6	6.0
60	9.4	64.1	11.8	34.8	4.7	10.7
50	8.9	73.0	11.5	46.3	6.8	17.5
40	7.7	80.7	10.7	57.0	11.3	28.8
30	7.8	88.5	10.2	67.2	14.2	43.0
20	4.6	93.1	11.6	78.8	15.4	58.4
10	4.7	97.8	11.5	90.3	15.6	74.0
0	2.2	100	9.7	100	26.0	100

college chemistry over those who have had only high-school chemistry, and of the latter over those who have had no chemistry for entrance. In the top 10 per cent we find 22.2 per cent of those with college chemistry, 5.7 per cent of those with only high-school chemistry, and 1.3 per cent of those without chemistry. At the 50-percentile the percentages are 73.0 per cent to 46.3 per cent to 17.5 per cent, that is, only one-sixth of those without chemistry for entrance are in the top half of the class, leaving five-sixths in the lower half of the class. In the bottom 10 per cent there are 2.2 per cent of those who have had college chemistry, 9.7 per cent of those with only high-school chemistry, and 26 per cent of those without any entering chemistry.

At the Naval Academy a mark of 4.00 is 100 per cent, 3.40 is "with distinction," 3.00 is considered "average," and below 2.50 is failure. Those who finished the course "with distinction" included 41.4 per cent of those who had had college chemistry, 16.7 per cent of those with only high-school chemistry, and only 4.0 per cent of those who had had no chemistry prior to entrance. In this horserace the odds are 1:2 that a man who has had college chemistry will win the race "with distinction," 3:1 for those who have had only high-school chemistry, and are long-shot odds of 15:1 for those who have had no chemistry. Above the "average" of 3.00 we find 79.0 per cent of those with college chemistry, 55.3 per cent of those with only high-school chemistry, and only 26 per cent of those without entering chemistry. The failures included 1.7 per cent of those with college chemistry, 5.8 per cent of those with only high-school chemistry, and 14.2 per cent of those without chemistry.

Usually the argument of those who believe that the year of physics or chemistry in the high schools is of no great advantage is that the effect of badly learned concepts outweighs the advantage of the earlier contact with science. This may be so in so far as course content is concerned, but an early beginning in proper methods of calculation and problem working, and in



laboratory work and its learning with the hands, ears, eyes, and mind is certainly an advantage.

One of the unasked-for results of the qualitative thinking of those who do not believe in the advantages of high-school chemistry is that many boards of control, listening to these fallacious arguments, have seen fit to drop physics and chemistry from the entrance requirements. Too many of our students thus come to us not only poorly prepared but without any interest in the sciences or in mathematics. The qualitative interest of juveniles in space ships and interplanetary travel is a poor substitute for a thorough grounding in algebra, trigonometry, physics, and chemistry.

CONCLUSION

This then must be our conclusion: There exists a definite correlation between a student's chance of success or failure in general college-chemistry courses and his previous preparation in chemistry in secondary schools. The chances of failure are three to four times as great for those who have not had previous chemistry as for those who present it upon entrance. The chance of passing the course "with distinction" as shown in Naval Academy data is nearly ten times as great for those who have had previous college chemistry as for those who have had no chemistry; and four times as great for those with high-school chemistry.

This is the quantitative or scientific approach. Let those who "believe but have no data," who "know from experience," who "feel sure but have no quantitative data," collect for themselves some data. Until they do, please let us have no more of this fallacious thinking that it makes little difference whether a student has or has not had previous training in the subject. Let us at least apply the scientific method to analyze that which is so important to elementary science courses in the colleges, and cease the qualitative approach.

HENRY WILLIAM STIEGEL—PIONEER IRON AND GLASS MAKER[1]

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AMONG the thousands of German immigrants to Pennsylvania in the eighteenth century there were few who contributed more to her economic development than Henry William Stiegel. Probably none was so colorful as this native of Cologne, who in the short span of three decades gained and lost a fortune, lived in baronial pomp, and still made significant contributions to two infant industries—the manufacture of iron and glass.

Iron was the source of the Stiegel fortune and his chief maintenance, but glass making was his love and the cause of both his fame and folly. He was a successful entrepreneur in both fields for a time, until his ambition to expand in new phases of glass manufacture, plus the squandering of money on estates and entertainment, caused his financial failure.

The scene of Stiegel's active career was Lancaster County, Pennsylvania, to which he came in 1752. He began work with Jacob Huber, an early ironmaster at Elizabeth Furnace, near Brickerville (*1*). He soon learned the business, married Huber's daughter Elizabeth, and succeeded Huber in the growing iron manufacture. Later he became co-owner and manager of another iron works at Charming Forge, located at Womelsdorf, Berks County.

The products of these operations—metal for fabrication in domestic and foreign plants, and finished products such as stoves, kettles, and later cannon balls—were sorely needed throughout the country.

An ironmaster in those days became a large landowner, mill operator, storekeeper, and in a true sense the chief figure in his community. His activities included the procurement of materials, the mining of ores, quarrying of limestone, timbering for lumber and charcoal. He was a merchant in materials and labor. Much of the latter came from indentured laborers and artisans. He planned and erected all manner of buildings from barns to private "castles."

Stiegel, in addition to the demands necessary to his station, was an artist, a contributor to music and the decorative arts. In glass he saw a product which could be of great practical and ornamental value. At Elizabeth Furnace he began experiments in glass making in 1763. At first, primitive forms of glass for bottles, windows, etc., were made. Then as the success of his venture and the skill of his craftsmen became apparent he saw the need for a new location.

To the nearby village of Manheim he went to erect his famous Glass House in 1765 and to foster the development of this little village. From here he supplied to the growing colonies much that was needed in household glassware, as well as beautiful vases, urns, and his famous enameled glass.

To the chemist, Stiegel's contributions were of importance not only because he developed the manufacturing arts themselves but also because he made chemical glassware to order. Some of this was used by the small but increasing number of doctors, druggists, and chemists, and some was sent to sugar makers in the West Indies.

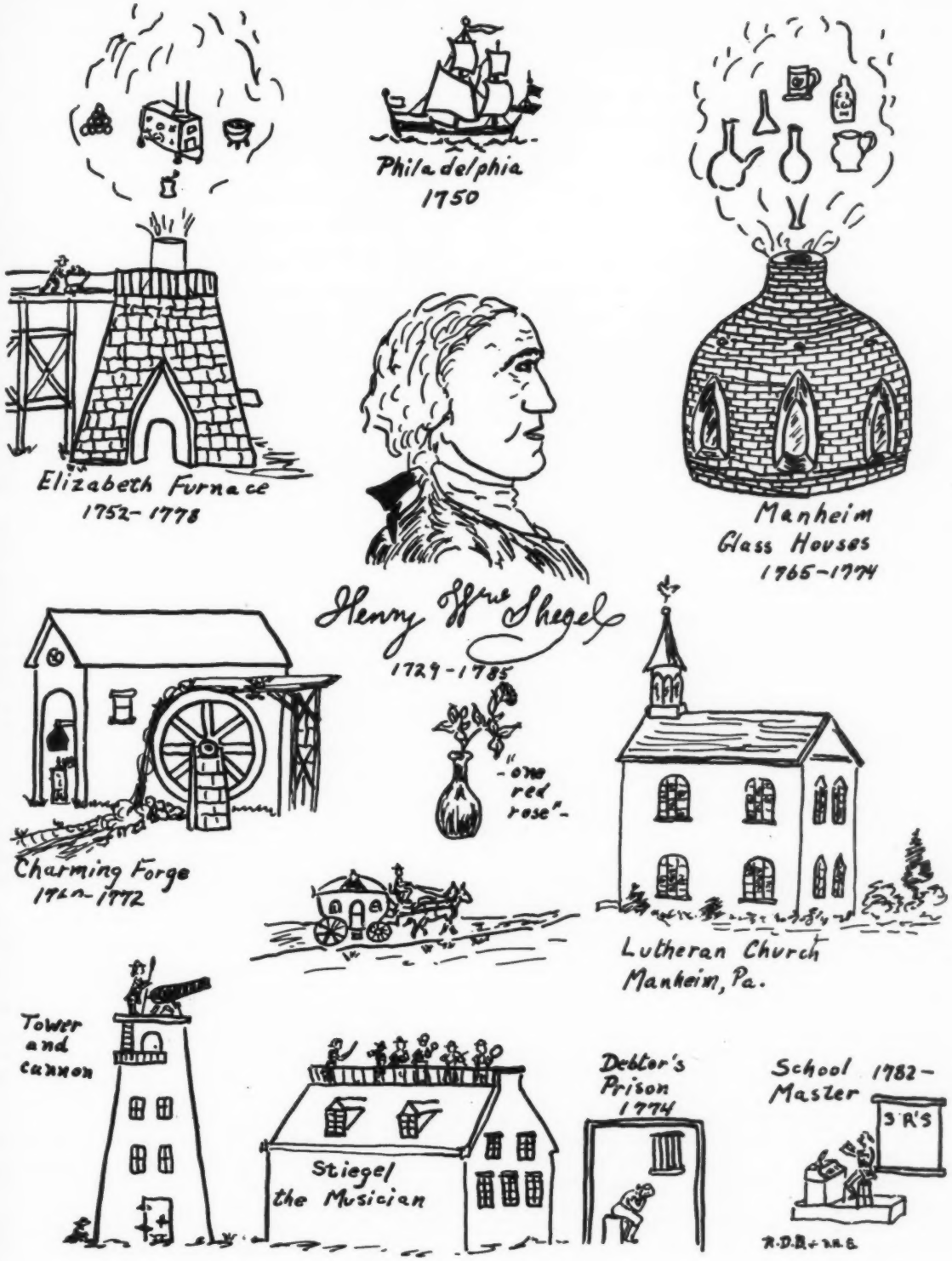
In still another field Stiegel's influence was to contribute both to posterity and to his individual fame. As a churchman Stiegel was active, first in his own community at Brickerville, later in his hey-day in Manheim. He was active in local church business, a delegate and counselor and a contributor of land for church building. This last connection in more recent years has resulted in far-famed publicity for both himself and Manheim.

In granting the land for t' e Lutheran Church in Manheim Stiegel simply requested in poetical sense the ground rent of "one red rose in the month of June if the same shall be lawfully demanded" (*2*). The specification of a rose as annual payment was not unusual. It was customary to state some such common article—for example, an ear of corn or something readily at hand as annual payment. Since 1896—over a century after his demise—this gift has been made to a Stiegel heir and the event has been widely heralded as the Festival of the Red Rose. This event inspired the writing of a popular novel "One Red Rose Forever" (*3*) whose chief character is the romantic "Baron" Stiegel. The revival of the actual practice to commemorate Stiegel's benefaction was due largely to the activites of Dr. J. H. Sieling, a young physician, who became so interested in the traditions surrounding Stiegel that he enthused increasing numbers of friends to participate in this festival.

There are many doubtful points in the Stiegel career which have not been explained, but let us first review the highlights of his activities from some of the accepted sources of information.

Late in the summer of 1750 Stiegel came to his adopted country together with 270 other German immigrants. With him came his widowed mother and his 11-year-old brother Anthony. The ship "Nancy," with Captain Thomas Coatem in command, arrived from Rotterdam after a seven-weeks' voyage at the port of Philadelphia on August 30. Some of Stiegel's

[1] Presented at the 122nd Meeting of the American Chemical Society, Atlantic City, New Jersey, September, 1952.


Elizabeth Furnace
1752-1778
Philadelphia
1750
Henry Wm Stiegel
1729-1785
Manheim
Glass Houses
1765-1774
Charming Forge
1770-1772
"one red rose"
Lutheran Church
Manheim, Pa.
Tower and cannon
Stiegel the Musician
Debtor's Prison
1774
School 1782-
Master
3 R's

descendants claim that he came with 20,000 pounds to invest in new enterprises on this side of the Atlantic. This is generally doubted because most of the passengers were poor, sturdy souls—many of them indentured servants who had to work for a number of years to pay for their passage. They came to the land of freedom and opportunity from the oppressed regions of the Rhineland.

Henry William Stiegel was born in Cologne, Germany, May 13, 1729. Details of his early life and schooling are unknown. Those who thought he was of royal blood have been unable to substantiate the claims. F. W. Hunter (*4*) who made the most extensive study on the life and work of Stiegel attempted to consult authorities on German royalty and could find no evidence for the baronial title, which was often given to him by his contemporaries. However, he must have been moderately well schooled, judging from his correspondence in both English and his native language. At the close of his career, when his business ventures had failed, he served as schoolmaster and music teacher in several small communities.

Stiegel's business contacts almost from his arrival in America were associated with two Philadelphia Quaker brothers, Charles and Alexander Stedman. In fact they may have owned the ship which brought Stiegel here, and their interests involved the traffic in immigrants, the sale of vast land holdings, and the financial backing of the primitive industries.

For the first two years in America we can only guess what Stiegel did. Perhaps he worked for the Stedmans, either in the Quaker City or on missions elsewhere. However, in 1752 we find him with his future father-in-law, Jacob Huber, at Elizabeth Furnace.

A COLONIAL IRON INDUSTRY

The region to which Henry Stiegel came to seek his fortune had already given promise to the long series of successful iron ventures which have marked its history. The Cornwall mines in Lebanon County just north of the red sandstone hills which border Elizabeth Furnace have been a continuous source of magnetic iron ore from 1742. They supplied the ores for the Cornwall Furnace (*5*) and for Elizabeth Furnace during the active years in which they were in blast. Jacob Huber had come to the northern section of Lancaster County in 1743 and several years later began the operation of this historic furnace.

When Stiegel came to Pennsylvania the frontier was just beyond the Blue Mountains on the north and not much farther west than the Susquehanna River. The entire population of the province was about 150,000 and this was concentrated largely in the southeastern section. Lancaster County was so constituted in 1729 as the result of a request from petitioners of northwest Chester County who sought a relief from long miles of travel to the county seat of Chester to conduct official business. It was the fourth county in Pennsylvania, following Philadelphia, Bucks, and Chester counties. As early as 1709 some Swiss Mennonites had migrated here. But expansion was under way, as newcomers arrived to take advantage of the opportunities in Penn's woods. The woods were extensive, as is evident from early estimates that 90 per cent of Pennsylvania was timber covered. Huge oaks, maples, birch, pine, hemlock, chestnut, and numerous other species of virgin timber blessed the land.

William Penn's hopes for developing his province had encouraged a planning board—The Free Society of Traders. Searches for raw materials and development of these into finished products were fostered. Grist mills, saw mills, tanneries, and paper mills, were among the first developments after land was cleared for homes and farming. In 1698 a report to Penn stated that iron mines near Durham, in Bucks County, were being guarded by a group of Shawnee Indians (*6*). Ten years later the mines were being worked.

Iron manufacture began in Pennsylvania with a bloomery forge erected by Thomas Rutter on Manatawny Creek in 1716 (*7*). Pennsylvania's first blast furnace was at Colebrookdale on Iron Stone Creek some four years later. Following soon after in the same region furnaces were erected at Reading and Warwick, and forges which were to become famous, Hopewell and Valley Forge.

The early furnaces were simple in operation and required only a few workers. A stone shaft, approximately 25 feet square at the base and tapering slightly to a height of perhaps 30 to 40 feet, constituted the external structure. (The earliest furnaces were somewhat smaller.) The interior was lined with firebrick or fine grained sandstone, the inside dimensions varying from three feet at the hearth to seven feet in the bosh. Air was supplied from a huge bellows operated by water power. It required only a half-dozen workers to operate the smaller furnaces, perhaps nine or ten for larger furnaces. To supply such a furnace with ore, which produced only two tons of pig iron every day, required but three or four miners working in open pits. However there was a larger force necessary to supply the wood and convert it into charcoal. Here a dozen men might be required. Seasonal conditions interferred with some or all of these operations and the best output from a furnace was only a few hundred tons of iron a year. In 1760 Pennsylvania produced 5000 tons from some 12 furnaces. This represented one-half of the total produced by all the American colonies.

At Elizabeth Furnace Stiegel began as a clerk and assistant to Huber. Within the year, however, in this delightful glen on a hilly slope, one mile north of Brickerville, he not only learned the fundamentals of iron making but won the heart of the ironmaster's daughter, Elizabeth, whom he married in November, 1752. In a few years, with his earnings and capital supplied by his Philadelphia friends, the Stedmans, he had acquired a partnership in the business and became its manager.

Stiegel and his partners were ambitious. Growing demands for iron required more raw materials. The 400 acres of land owned by Huber was insufficient to

Elizabeth Furnace, Home of Henry William Stiegel

supply wood enough for the charcoal needs of the furnace. So within two years the estate grew in size to 10,454 acres. The clearing of the land not only furnished wood for charcoal and timber for construction but it also provided new areas for cultivation of crops. Lancaster County, well blessed with limestone, has since become known as the garden spot of Pennsylvania because of its rich soil. Pioneers like Huber, Stiegel, and their associates in iron production were making it possible for more of the sturdy German settlers to come to this region. Iron was but one step, albeit a very important one, in the development of this lush agricultural area.

Some idea of the charcoal used by an average furnace of the late eighteenth century is obtained from the writings of Henry Drinker, who in 1786 estimated that one furnace required 800 bushels of charcoal every 24 hours. Estimating (*8*) that this could be supplied from twenty cords of wood, which was almost the average cut from an acre of woodland, it would require almost 250 acres of forest section to supply charcoal for a possible eight months of blast furnace operation per year.

Dr. Herbert H. Beck, dean of Lancaster County historians, guided the author over historic spots in the Furnace Hills where Stiegel operated. At the Elizabeth Furnace estate we saw the strong buildings of native sandstone, which bear evidence of both the original workmanship and the care which has been exerted in maintenance over the long years. The original home of Jacob Huber (built 1746) in which Stiegel was married is still in good repair, although not used as a residence. More imposing are the homes of the later ironmasters, the Stiegel home of about 1760 and the larger mansion of Robert Coleman built sometime after the latter acquired the property during the Revolution. Behind the Stiegel home of two and one-half stories is a small annex used as an office, to which is attached a long stone barnlike structure which housed a store and warehouses. This latter was used as a barracks for Hessian prisoners who worked for a time under Stiegel's direction. The belfry atop the barracks was used to summon the workers on the iron plantation to meals and assemblies. Of interest to the modern engineer as well as the antiquarian is a small building which was used as the ice house. Its square stone walls, eight feet on a side, were capped with a relatively steep, peaked roof of wooden shingles.

Tangible evidence of the iron work produced by Stiegel's craftsmen remains in the shape of cast-iron stoves. Cylindrical, or cannon stoves, and the boxlike six- and ten-plate stoves with ornamented sides show that advances were made in stove design and decoration. We saw one in the Coleman mansion at Elizabeth Furnace made by Stiegel, and another of later date. There are others in Pennsylvania museums at Reading, Doylestown, and Hershey. In the ten-plate stove there was an internal oven with a side door. This oven is claimed (*9*) to have been the first cooking oven apart from the primitive fireplace and attached bake oven. This type of stove sold for something over five pounds, depending on its size and ornamentation. We wonder what obscure iron maker, perhaps urged by his long suffering wife, first attempted this advance to ease the problems of home cooking. Also from Elizabeth Furnace came cast-iron pots for the soap and apple-butter making of the farmers, kettles for the hearth, cannon balls for the wars against the French and Indians and later for the War of Independence.

Steigel was also a partner and manager in another important iron works. This was a forge northeast of Brickerville along the Tupehocken Creek, near Womels-

Specimens of Stiegel Iron Work

Upper Left: Elizabeth Furnace Stove-Plate
Lower Left: A Stiegel Cannon Stove, Owned by the James Spear Heating Company of Philadelphia
Right: A Stiegel Stove in the Museum of the Berks County Historical Society at Reading

dorf, in Berks County. It was only a little more than ten miles as the crow flies but nearer 20 over the hill on the primitive roads. Romantically, Stiegel named it Charming Forge, being impressed by its pleasant location. The product of Charming Forge was refined iron—wrought bar iron—made from the pig iron trundled by horse and ox carts from Elizabeth Furnace and other sources. The bar iron was malleable and from it could be made axes, farm tools, plough heads; spikes and nails for building; axles and tires for the wheel rims of the massive Conestoga wagons—another Lancaster product which became the ark of transportation for many settlers pushing westward.

GLASS MAKING

Steigel was not the first to make glass in Pennsylvania. Nor did his several glass houses continue in very prolonged operation. However he had a flare for the artistic and his Manheim glass works were probably the first in America to make beautiful flint glass and colored enameled glass.

His ventures in glass making were both his joy and his folly. What started this successful young ironmaster to embark in a new field of industry? First we think it was in answer to a demand which was widespread and evident. Window glass and bottles were needed whenever the settler got beyond his crude beginnings. So these were the products which Stiegel and other early glass makers produced first.

In the history of glass making it was not until the eighteenth century that window glass became available to the average man. Even then the small panes offered at high prices were uneven in thickness and produced distorted views. Stories, perhaps with some truth, have been told of loads of glass which were hijacked as they were being transported inland to demanding housewives.

There are certain similarities in iron and glass making, perhaps more in their primitive stages of production. Slag, a by-product of the blast furnace, is essentially an impure glass. Such thoughts might have prompted Stiegel in 1763, at Elizabeth Furnace, to begin experiments in glass making. He may have discovered among his workers some who had been glass makers before coming to America. It has also been surmised that Stiegel himself knew something about the glass industry at first hand from his youth in Cologne. The dates which Hunter gives for work at this first glass house are from September 18, 1763, to November 11, 1765.

Soon his success in the glass venture urged him to select a new location, where glass manufacture could be the chief industry. His partners, the Stedman brothers, had acquired 729 acres of good level pasture land in Rapho Township, about eight miles southwest of Elizabeth Furnace. Stiegel became holder of one third of this land and together with Charles and Alexander Stedman make plans for the settling of the town of Manheim. There were already 118 families, chiefly German and Swiss farmers, in the township in 1756.

Here in Manheim Stiegel erected two glass houses. The first was begun October 4, 1764 and was operated from October 27, 1765, to April 20, 1769. The second factory, begun in 1768, is said to have been 90 feet high, with dome-shaped walls of red brick imported from England. The glass works operated intermittently, closing down in the warm summer months, when the glass blowers would leave to work on farms or migrate elsewhere.

Additions were made to buildings during 1771, 1772, and 1773, but in May, 1774, after a continued decrease in glass sales, the business was leased to a company of Smith and Simund. This firm evidently did not function very long and many have been assigned only to help in sales of remaining stock. Altogether Stiegel's contact with glass manufacture covered only about ten years. During this interval he was busy planning buildings, securing raw materials, engaging glass blowers, and selling his wares.

The ingredients for glass making are not too complex. Quartz or silica is widely and abundantly found in nature and could be obtained locally. Some potash was probably extracted from wood ashes in the region, and more obtained later in exchange for finished glassware in the seaboard cities of Boston, New York, and Philadelphia. Lead oxides, both litharge and red lead, for his flint glass were obtained from both domestic and foreign markets. Limestone to furnish lime was no great problem. Elizabeth Furnace had its own limestone quarry within a few miles. Metal oxides for color—chromium, cobalt, copper, and iron—could all be supplied from colonial sources. There is cobalt, for example, in the Cornwall iron deposits, but it is doubtful whether its existence was known at the time. Pyrolusite, the manganese dioxide used both as a clarifier to counteract traces of iron or in larger amounts to produce an amethyst color, was imported.

At first the Manheim production of glass was also strictly utilitarian—bottles, window glass, common mugs, tumblers, and household necessities. Later, as the number of workmen and their talents increased, came the era of artistic specialties. Stiegel made at least one trip abroad to hire glass blowers and to learn foreign glass methods. This was late in 1763. Since much of his later glassware follows the so-called Bristol technique, it is believed that much of his time was spent visiting glass works there.

On July 1, 1772, the name "The American Flint Glass Manufactory" was announced to designate the Manheim plant. Stiegel was advertising his products through dealers in many of the larger cities and towns in the colonies. He appealed to the patriotic spirit of the public, as is evidenced in this quotation from an advertisement in the *New York Journal or the General Advertiser* of January 14, 21, 28, and February 4, 1773: "...he hopes for the encouragement of those who wish well to the establishment of manufacturers on this continent: and that the glass he offers to the public will be found to rival that which is imported and sold at lower prices."

In another advertisement in the *New York Gazette and Weekly Mercury* of February 8, 15, 22, and March 1, 1773, Stiegel states—after enumerating various types and sizes of decanters, tumblers, wine glasses, rounds and phials for doctors, ink bottles, etc.—"Orders taken for all kinds of glasses for chymical or other uses, agreeable to patterns. It is expected that all friends to American manufacturers will do their utmost in promoting this."

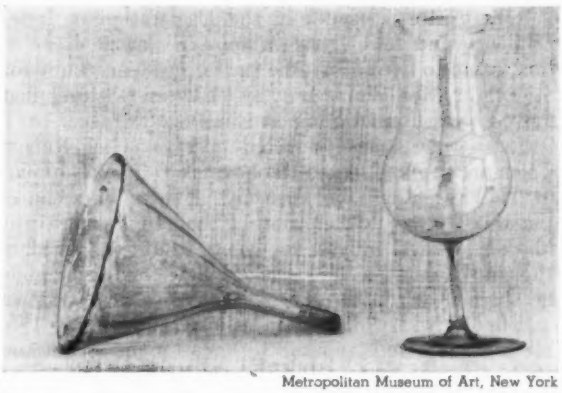
Considering the quality of his work it is surprising that his dealers in Boston thought it necessary to offer Stiegel glass to the public as "imported."

Window glass from the Stiegel works was made by both the "crown method" (spinning an opened bubble of glass into a flat circular disc) and the "sheet" method (flattening, under the influence of heat, a pane of glass first blown in a cylinder and cut lengthwise by a diamond). All other products may be considered under four general types of glassware. First, there was blown glassware which involved no mold; second, the type which shows ribbed or molded surfaces and required the use of a small open-topped design mold; third, pieces which were engraved by diamond point or metal wheel using emery; fourth, the glassware decorated and then baked with vitrifiable colored enamels.

This latter ornate type of product was made late in the life of the Manheim glass factory. It is gaily colored in designs which have inspired the revival of an art often called Pennsylvania Dutch. Here the Stiegel artists were producing on glass decorations similar to those which were the vogue in pottery, wood, and painted tinware. In Manheim the author was privileged to view a beautiful collection of authentic Stiegel glass at the home of Mr. and Mrs. Miles Keiffer on Market Square, only a few doors from the building which was the Stiegel mansion.

This paper cannot trace the intricacies of the Stiegel accounts, business deals, and later failures. A career which was marked at intervals with extravagant purchases—homes, "castles," tapestries, musical instruments, showy coach, and fine horses—plus extensive land holdings and business expansion resulted in financial obligations which could not be met. Stiegel's donations and benevolences had established his generosity and public spirit, but there were also instances of runaway servants, embittered workmen, and merchants demanding payment on all sides. "In the end," Hunter states, "he put all his financial eggs into this glass basket." And to continue the analogy one could say that it took fancy stepping to delay the crash.

Stiegel even tried a widely advertised public lottery to help raise funds for his glass industry. This device was often used in colonial days as it is today in many Latin American countries. But the times were poor and Stiegel could not get much interest in his schemes. He estimated that his entire holdings in land, houses, and furnaces might be worth almost 20,000 pounds, which would amply settle his accumulated debts of about 10,400 pounds. He wanted to sell all except the glass houses, but foreclosures and lawsuits haunted him.



Metropolitan Museum of Art, New York

Examples of Chemical Glassware from the Hunter Collection of Stiegel Glass

Even his friends could not keep him from a short period of "token" imprisonment for debt in 1774. However, release from custody came to Stiegel the day before Christmas at a special Council meeting presided over by the Honorable John Penn, Esq., Governor.

Stiegel's last years were active and useful to his countrymen, although his station in life was much less dignified and his letters were humble and plaintive. His mansion at Manheim was acquired by leading figures of the Revolutionary period. He was indebted to John Dickinson, prominent Philadelphia attorney and member of the continental Congress and to him wrote many letters which recount the state of his accounts and also the depth of his feelings. These letters are in the files of the Pennsylvania Historical Society in Philadelphia and are the source of much of the information of the authorities quoted.

Philadelphia Museum of Art

Enameled Tumblers and Drug Bottle of Stiegel Manufacture. Manheim, ca. 1773

Robert Morris, signer of the Declaration of Independence and termed the "financier" of the Revolution, came to live for a time in the residence built for Stiegel in Manheim. But by this time Stiegel had moved to his former home at Elizabeth Furnace.

For a time Stiegel directed operations for Robert Coleman who had leased the Furnace to make supplies for the struggling colonial army. When the scene of the war shifted farther south Elizabeth Furnace was idle and Stiegel moved elsewhere. Later he lived in the church parsonage at Brickerville; then for a while he stayed with his brother Anthony in Schaefferstown and finally with George Ege at Charming Forge. In his last years he is reputed to have taught school and clerked at some of the iron works of his friends and relatives.

Henry William Stiegel died January 10, 1785, at the Ege home near Womelsdorf. There is no certainty as to where he is buried.

The visitor to Manheim will find at the east end of Market Square a dignified plaque in blue and gold honoring "Baron Stiegel." Erected in very recent years by the Pennsylvania Historical Commission it would seem to give official approval to the title frequently ascribed to Henry William Stiegel. Apparently the only place the title was printed during Stiegel's life was on some of the stove plates made at Elizabeth Furnace. One such inscription ran:

Baron Stiegel ist der Mann
Der die Oefen giessen kann.

No doubt Stiegel was so honored by his workmen and the provincial population, and needless to say he liked the title and tried to live up to it. However, no signature of his ever has been found which puts his claim to title in writing. As Dr. Beck states, "the very mystery of his ancestry, like the uncertainty of his place of burial, only intensifies the glamor of the picturesque and colorful figure of Stiegel. His fame is well established by his glassware, by the firm traditions of the baronial pomp of his career, and by the churchyard, which he gave to the Manheim Lutherans for one red rose."

ACKNOWLEDGMENTS

The author wishes to acknowledge the help and hospitality of Dr. Herbert H. Beck and Mr. and Mrs. Miles Kieffer in rendering assistance in connection with this writing. He appreciates also permission granted by Mr. George L. Heiges of Manheim, and officials of the Philadelphia Museum of Art to reproduce illustrations shown. An explanation of the sketch made by the author depiciting events in Stiegel's career is in order. The several drawings surrounding the central head are, we hope, self-explanatory. Stiegel's signature has been copied and his "likeness" drawn from a head depicted on one of the Stiegel stove plates. Actually there is no known picture of Stiegel extant, so indulgence is requested for artistic (?) liberty. Mr. D. N. Stehly assisted by retracing the sketch.

Mr. C. S. Brinton, of Haddonfield, New Jersey, was kind enough to read this paper and offer several valuable comments. The production figures for colonial iron production in Pennsylvania were altered, at his suggestion, from a lower estimate.

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CLARA MARIE DE MILT

As this issue goes to press we have just learned of the sudden death of one of our most respected and esteemed contributors, Dr. Clara de Milt, head of the Department of Chemistry of Sophie Newcomb College of Tulane University. She had a wide reputation as a teacher and historian of chemistry, and her sound judgment and good humor could always be depended on. She will be greatly missed by her many friends.

LACQUER COLOR FILTERS FOR QUALITATIVE FLAME TESTS[1]

JOHN A. BROWN
Georgia Institute of Technology, Atlanta, Georgia

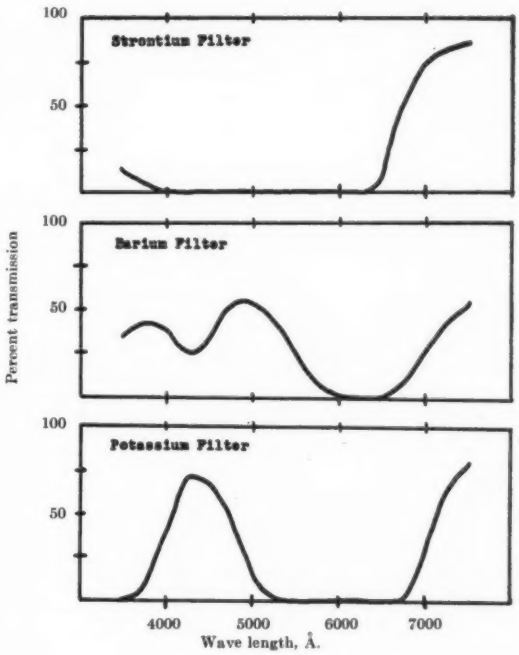
THIS paper describes the manufacture of certain transparent colored lacquers and their use as color filters in the semispectroscopic detection of the alkaline metals.

In theory, it should be easy to see one colored flame in the presence of another by observing the combined flame thru a color filter which transmits the spectral lines due to one flame and blocks those of the other. This principle is employed in the familiar flame test for potassium, wherein a cobalt glass is used to filter out the yellow light of sodium. The filters described herein extend this application to include barium and strontium.

An examination of the visible flame spectra of barium and strontium salts reveals that each has bright lines in a region of the spectrum where the other has none. (This is not true of calcium, all of whose bright lines are nearly duplicated by either barium or strontium.) Therefore, a color filter which blocks red and yellow light, but passes green, should enable one looking through it to see a barium flame, but not a strontium flame. Similarly, a filter passing red, but blocking yellow and green, should enable one to see a strontium flame, but not a barium flame.

For the past several terms, we have been using such filters as the sole means of detection of barium, strontium, sodium, and potassium in an abbreviated course in qualitative analysis at Georgia Tech. The analytical procedure is simple: First barium and strontium are separated from sodium and potassium by precipitation of the barium and strontium with $(NH_4)_2CO_3$. Then the precipitate, after washing, is dissolved in a few drops of concentrated HCl. A platinum wire is dipped into this solution, and held in the hottest part of a Bunsen flame and the resulting colored flame is observed first through a red-transmitting "Strontium Filter," and then through a green-transmitting "Barium Filter." Similarly, the sodium-potassium flame is observed with the naked eye for sodium, and through a red-and-blue-transmitting filter for potassium. If any flame is visible through the "Strontium Filter" strontium is reported; if any flame is visible through the "Barium Filter" barium is reported; and similarly for sodium and potassium. Actually, since the filters are seldom perfect, a glimmer of the wrong flame may sometimes be seen; but these glimmers can easily be distinguished from the strong, steady flames of positive tests.

[1] Presented to the Joint Meeting of the Georgia Academy of Science and the Georgia Section of the American Chemical Society, Atlanta, Georgia, April 18–19, 1952.



In the case of barium and strontium, the strength of the flame is not dependent upon the concentration of the metal ion, but is reasonably constant for any concentration which is sufficient to yield a precipitate with

Preparation of the Filters[a]

Lacquer Base	
Acetone	200 ml.
5–6 Sec. nitrocellulose	30 gm.
Cellosolve	60 ml.
Methyl amyl acetate	200 ml.
Barium Filter	
Lacquer base	45 ml.
Dye soln. (140 mg. DuPont Luxol Green BL in 5 ml. 95% ethanol)	5 ml.
Calco acetate green 2B	210 mg.
Potassium Filter	
Lacquer base	50 ml.
Calcozine pure blue BO	460 mg.
Strontium Filter	
Lacquer base	50 ml.
Cellanthrene brilliant blue (DuPont)	70 mg.
Calcozine green V	20 mg.
Calco acetate red	75 mg.
Calcozine violet ex. conc.	50 mg.
Calco diazo black	115 mg.

[a] Formulas courtesy of The Sunfilm Corp.

$(NH_4)_2CO_3$, since the precipitate obtained is dissolved in the minimum amount of HCl, leading to a reasonably constant concentration in the flame solution. The sodium and potassium flames do vary in strength with the concentration since no precipitate is ever obtained, and this variation may be used to estimate concentrations.

Each student prepares his own set of filters by painting inch-square spots of special lacquers onto glass plates and allowing them to dry.

The lacquers themselves are prepared by coloring nitrocellulose solutions with appropriate textile dyes, as shown in the table. The nitrocellulose is rather slow to dissolve, and the mixture should be stirred in a closed vessel—or better, shaken—until the solution is completely smooth. Then the dyes are added to portions of the lacquer base and agitated until solution is complete. Since most organic dyes are somewhat fugitive to light, the finished lacquers should be stored in brown bottles and the finished filters kept in the dark when not in actual use.

The figure shows the transmission spectra of typical lacquer films on glass. The exact transmission values will, of course, vary somewhat depending on the thickness of individual filters. The filters used to make these spectra were entirely satisfactory on actual flame tests.

ACKNOWLEDGMENT

The author wishes to express his appreciation to Dr. W. C. Carter of the Georgia Tech. Textile School for his help in suggesting likely dyes.

A CLOSET FOR MEASURING CONTINUOUS DRYING RATES

ERNEST E. HART and HOWARD L. DUNLAP
Ohio University, Athens, Ohio

It is frequently a problem in the laboratory to devise means and procedures for illustrating a principle where the available time can yield worth-while data. Laboratory experiments in unit operations are especially difficult in this respect, and the drying rate of materials is typical of such an operation. The procedure should be of such a nature that not more than one period need be used to assemble the apparatus and carry out a practice run. Then the following period or periods can be utilized for securing data with little interruption.

Close-up of Balance and Air-ducts

The following is a description of the arrangement of a Stroke drying closet, No. 51, which has worked quite satisfactorily. It was thought that the suggestion might prove useful to others.

The closet has a regulated steam-heating unit and is equipped with an air-circulating fan. The amount of air recirculation can be regulated, and the inlet and outlet air ducts are each fitted with wet and dry bulb thermometers. A rigid stand was mounted on top of the drying closet and on this stand was placed a beam pan-balance capable of carrying ten kilograms. The pan or tray for drying material was suspended from a beam centered on a pivot over one end of the beam of the weighing balance, leaving the other end of the balance beam for the load weights. Over the center of the tray beam a smaller pan was mounted to receive weights for determining the evaporation loss during the time interval. As evaporation proceeds more

weights are required in this pan to balance the load weights in the other pan. The tray is connected by four rods from the two cross beams outside the drying closet. The four posts extending through the top of the drying closet were each made in two sections, joined by sockets and pins. This permitted the removal of the tray. Movable weights were placed on the rigid cross beams outside the drying closet; by moving these the four tray posts can be made to swing freely in the holes through the drying-closet top.

All the materials used in the construction are aluminum rods and bars; the bottom of the aluminum tray was reinforced by two aluminum bars to prevent sagging. The sensitivity in weighing ranged from 0.2 g. for 1 kg. to 0.8 g. for 10 kg.

The procedure consists of first adjusting the load so that the tray posts swing freely through the opening in the top of the closet. The air-circulating fan is started and the temperature is raised to the desired point and allowed to become constant. The fan is then stopped long enough to weigh the load and have it properly balanced. The circulating fan is again started and allowed to run for the desired time interval whereupon the fan is again stopped. Weights are added to the evaporation-loss pan to obtain the evaporation for the time interval. The drying closet is kept closed until the end of the experiment.

The tables show a part of the experimental data from a 10-pound load of precipitated calcium carbonate on a tray measuring 3 feet on a side, and spread to a depth of about $^3/_8$ of an inch. The steam pressure in the coils was kept at about 15 pounds. Table 1 illustrates drying at the constant-rate period.

TABLE 1
Constant Drying-Rate Period

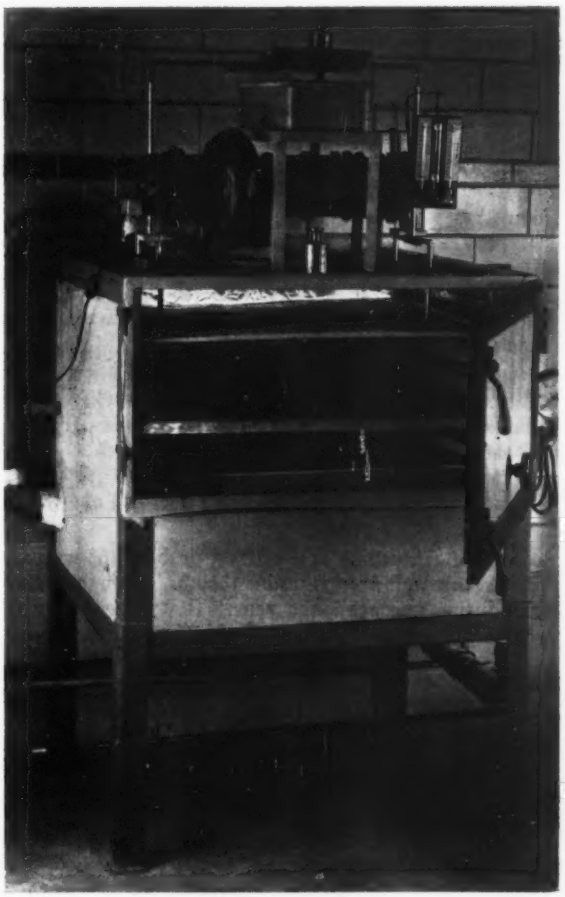
		—Air, ° F.— In	Out	—Humidity— In	Out	Time, min.	Wt. lost, g.	Temp. of closet
1	Wet	58.4	64.0	0.0067	0.008	15	104	156
	Dry	76.0	85.1					
2	Wet	59.2	64.2	0.006	0.007	15	111	160
	Dry	76.0	86.1					
3	Wet	59.0	64.2	0.006	0.007	15	108	155
	Dry	76.0	87.1					
4	Wet	60.0	64.4	0.007	0.008	15	119	160
	Dry	77.0	87.0					

At the beginning of the next laboratory period, five days later, the tray had lost 555 g. when again brought up to drying conditions approximating the previous laboratory period, but it was still within the falling-rate period, as Table 2 shows.

For the falling-rate periods, three 15-minute periods are combined into the 45-minute period. When weight loss per time interval is plotted against the time of drying, an almost straight horizontal line is obtained. A consistent curve is obtained from the falling-rate data in Table 2.

TABLE 2
Falling Drying-Rate Period

		—Air, ° F.— In	Out	—Humidity— In	Out	Time, min.	Wt. lost, g.	Temp. of closet
1	Wet	64.1	68.2	0.009	0.011	45	84	157
	Dry	78.1	88.0					
2	Wet	65.1	69.5	0.009	0.011	45	56	160
	Dry	78.4	90.0					
3	Wet	65.5	69.1	0.010	0.011	45	42	161
	Dry	80.0	90.0					



Complete Drying Closet

THREE SCHEDULE PATTERNS IN FRESHMAN CHEMISTRY TEACHING

DAVID J. BLICK
University of Connecticut, Storrs, Connecticut

For many years the course pattern in general chemistry and in most other science courses involving laboratory at the University of Connecticut was two one-hour lecture periods and two two-hour laboratory periods per week for four academic credits. The origin of this pattern is not clear, but it is evident that it was used early in the history of the school and that by 1903 it had become well established (*1*, *2*). An examination of subsequent catalogues showed that this pattern was strictly adhered to until the fall of 1946. A review of catalogues of other colleges indicated that the amount of time devoted to lecture and laboratory in general chemistry varied considerably. Furthermore, a search of the literature revealed that very few studies have been made concerning the effect of varying amounts of laboratory and class work on achievement in general chemistry (*3*, *4*, *5*).

In view of the above, an investigation was begun in an attempt to determine the relative effectiveness of three different schedule patterns in the teaching of general chemistry at the University of Connecticut. The patterns chosen were as follows:

Pattern A. Two one-hour lecture periods per week and two two-hour laboratory periods per week.

Pattern B. Three one-hour lecture periods per week and one three-hour laboratory period per week.

Pattern C. Two one-hour lecture periods per week, one one-hour recitation period per week, and one three-hour laboratory period per week.

Because of scheduling and staffing problems it was impossible to teach groups under the three schedule patterns during one academic year. It was necessary to try one pattern each year, thus making the study extend over a three-year period. At the end of each academic year, the students were given a standardized A. C. S. Cooperative Chemistry Test for College Students. A thorough description of the preparation and standardization of these tests is given by Ashford (*6*). Furthermore, the coefficient of correlation between Form 1946 and the teacher-constructed mid-term examination used in the above classes was found to be 0.71 (N = 121). This is additional evidence of the validity of the standardized examinations for the purposes for which they were used in this study. Each examination consisted of the following parts: General Knowledge and Information; Application of Principles; Quantitative Application of Principles; Scientific Method; and Laboratory Technique. While it is recognized that many outcomes of laboratory work cannot be measured by paper and pencil tests, the laboratory section of the examination is considered by test experts and instructors to be defensible (*7*).

The data used in this study are shown in Tables 1, 2, and 3. The following additional information for each student was obtained: sex, scholastic aptitude test score, and age. All students involved were pre-science and pre-engineering majors with comparable backgrounds in secondary school science and mathematics.

TABLE 1
Results for the Three Patterns

	A	*B*	*C*
N	95	148	161
High	88	113	113
Q_3	64	69	75
Median	46	54	60
Q_1	39	42	48
Low	26	19	27
Mean	50.83	56.25	61.93
Standard Deviation	15.16	18.99	19.40

TABLE 2
Results on the Scientific Method Section of Test

	A	*B*	*C*
N	95	148	161
Mean	11.36	14.03	16.48
S. D.	4.98	5.24	5.36

TABLE 3
Results on the Laboratory Technique Section of Test

	A	*B*	*C*
N	95	148	161
Mean	3.87	3.40	3.24
S. D.	1.54	1.93	2.17

A preliminary analysis of the data indicated that significant differences existed between the three patterns. Since these differences might be due to variations in the three groups of students in age and scholastic aptitude, the following values were computed by use of the method of multiple regression in order to determine the relative importance of each factor (*8*). Age, scholastic aptitude score, and chemistry test score are represented by X, Z, and Y, respectively.

$$^{b}Y_{xz} = -0.472 \qquad ^{b}Y_{zx} = 0.185$$
$$R\,(\text{calc.}) = 0.32 \qquad R_{0.01} = 0.21$$

Since the calculated R is significant at the one-per cent level, the sum of squares due to regression is partitioned

into that due to age and that due to scholastic aptitude. An analysis of variance (Table 4) is used to test the significance of each factor. It is obvious from the calculated F values that the regression due to scholastic aptitude is very significant while the regression due to age is not significant. Hence the predictor scholastic aptitude will be used in matching the groups by means of the Neyman-Johnson Technique (*9*, *10*). This method of matching, using matched subgroups instead of individuals, has certain advantages over traditional methods. It is more likely to produce samples which are more representative of the original population, and it prevents loss of much valuable information.

TABLE 4

Source of variation	*Sum of squares*	*Degrees of freedom*	*Mean square*	F	$F_{0.01}$
Age	592	1	592	2.04	6.76
Scholastic aptitude	6,726	1	6726	23.20	6.76
Errors of estimate	63,874	218	290		
Total	71,192	220			

The results of the analysis of subgroups equated on the basis of scholastic aptitude are given in Table 5.

TABLE 5

Subgroup means of schedule patterns	t *(calc.)*	*Interpretation* $t_{0.01} = 2.58$
A versus B $(B > A)$	3.54	Diff. of means statistically significant in favor of B.
B versus C $(C > B)$	2.30	Diff. of means not statistically significant.
A versus C $(C > A)$	6.73	Diff. of means statistically significant in favor of C.

The important findings are: (1) Student achievement under Pattern B was greater than under Pattern A, as measured by the test used in this study. This finding is in accord with what might have been predicted. The students under Pattern B had 30 hours more of lecture per year, and they were required to prepare for class three times a week instead of two. It would seem, therefore, that the instructor could cover the subject matter more slowly and thoroughly. It is reasonable to expect that their achievement would be higher. The data indicate that it was significantly higher.

(2) Student achievement under Pattern C was higher than under Patterns A or B. However, the difference in achievement between Pattern B and Pattern C is not statistically significant at the one-per cent level. The difference in achievement between Pattern A and Pattern C is statistically significant in favor of Pattern C. It must be borne in mind that the success of Pattern C may be largely dependent upon the quality of graduate assistants available to conduct recitation sections.

(3) Student achievement in the laboratory as measured by the test used was slightly lower under Patterns B and C than under Pattern A. Student achievement in the laboratory as judged by the instructor and assistants in terms of written reports and of results on qualitative analysis unknowns was essentially the same under the three patterns. Since the students under Pattern B and Pattern C had one hour less of scheduled laboratory time per week, their achievement might be expected to be lower than was indicated by the data. However, the condensation of the two two-hour laboratory periods into one three-hour period did not mean that the student actually lost one hour of working time in the laboratory. The time spent in cleaning up at the end of the first period and in setting up the apparatus again the second period in the week as in Pattern A is eliminated in Patterns B and C. The actual loss of working time was probably not more than 30 minutes.

In interpreting the findings of a study of this type, there are several limitations which must be clearly recognized. In any study involving a large number of people over a period of three years, it is impossible to control many factors which might conceivably influence the findings. Also, this study involved only one university with the writer teaching the classes and graduate assistants conducting the laboratory and recitation sections involved. Therefore care must be exercised not to apply the findings to another population unless there is evidence to indicate that the populations may be considered similar.

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Proceedings of the

PACIFIC SOUTHWEST ASSOCIATION OF CHEMISTRY TEACHERS

PLASMA EXPANDERS[1]

ROBERT T. MERRICK
Don Baxter, Inc., Glendale, California

THE administration of blood or plasma to a patient in shock is a complex problem. We are all familiar with the elaborate precautions taken in the preparation and handling of our food. Yet, compare this to the care necessary in preparing and handling a material which is to be injected directly into the blood stream of a patient.

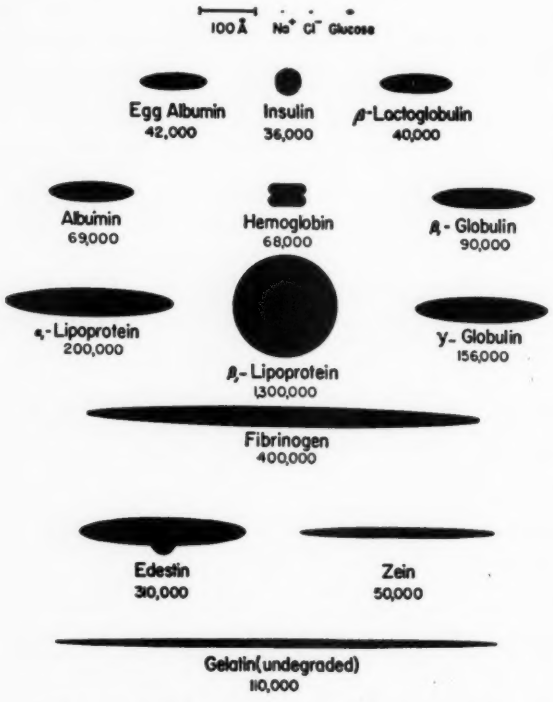


Figure 1. Molecular Shape and Size of Various Proteins[2]

Blood is collected for transfusion purposes by drawing it into a sterile bottle containing an anticoagulant solution. This mixture is then refrigerated and may be stored up to 21 days. Laboratory tests classify blood into groups according to blood types and Rh factors. No two bloods are exactly alike, however, and reactions sometimes occur due to small differences between the blood of the donor and that of a recipient. Reactions may also be caused by typing errors or by contaminated blood.

If the patient does not require the oxygen-carrying capacity of blood, a plasma transfusion may be preferable to a blood transfusion. Liquid plasma may be stored for two years. Irradiation of the plasma by ultraviolet light appears to have reduced the incidence of homologous serum jaundice caused by plasma injections. Lyophilized or freeze-dried plasma may be stored for 5 years, but the dry plasma and distilled water to reconstitute it before use require twice as much shipping and storage space as liquid plasma. All plasma transfusions are expensive and the supply of plasma is limited.

In view of the possibilities of atomic warfare, Dr. DeGowin, an authority on blood and blood transfusions, has said:[3]

> From data accumulated at Nagasaki and Hiroshima it has been estimated that five million pints of blood would be required in three weeks to treat the casualties resulting from ten atomic bombs dropped simultaneously on as many cities in this country. This is approximately one-half the amount collected by the Red Cross in this country during the entire war.

As you can see, we are in serious need of some substitute for blood or plasma.

A true blood substitute, that is, an oxygen-carrying material, is probably many years in the future. The so-called plasma substitutes do not have all of the properties of plasma, and for this reason they are frequently referred to as plasma volume expanders or plasma expanders.

An effective plasma expander should resemble albumin (Figure 1), the material in plasma responsible for most of its osmotic effect. The ideal plasma substitute should be:

1. Equivalent in colloidal osmotic pressure to normal blood plasma.
2. Stable during storage at all temperatures.
3. Fluid in cold climates.

[1] Presented before the Southern California Section of the Pacific Southwest Association of Chemistry Teachers on May 23, 1952, at Pasadena City College, Pasadena, California.

[2] ONCLEY, J. L., "Symposium on Nutrition," Charles C. Thomas, Springfield, Illinois, 1950, Vol. II, p. 3.

[3] DEGOWIN, E. L., *Surgery*, **28**, 121 (July, 1950).

4. Sterile.
5. Nonpyrogenic (that is, free of fever-producing contaminants of bacterial origin).
6. Nonantigenic (that is, free of antibody-forming substances).
7. Constant and uniform in composition.
8. Free of immediate or delayed reactions or damage to the recipient.
9. Completely excreted from the body, after it has served its purpose.
10. Free of interference with blood typing or blood administration.
11. Easily manufactured from plentiful and inexpensive raw material.

Many materials have been used as plasma substitutes or expanders. One of the first of these was a 2 per cent solution of gum acacia. This solution, widely used in 1937 and 1938, fell into disuse when it was found to be stored in the various vital organs of the body. In recent years many other materials have been used. Pectin, a methoxylated galacturonic anhydride occurring widely in plant tissues (Figure 2), is effective as a

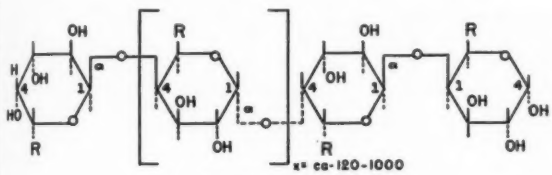


Figure 2. Pectin

plasma expander. However, the solution is not stable at a pH above 4.2 and must be mixed with a buffer solution before use. Crystallized bovine albumin, polyglucose, animal plasma, and an okra extract also have been used.

Recent work on plasma expanders has been concentrated on dextran, polyvinylpyrrolidone, and gelatin solutions. Dextran is a carbohydrate material (Figure 3) formed by the action of a bacterium, *Leuconostoc mesenteroides*, on sucrose.

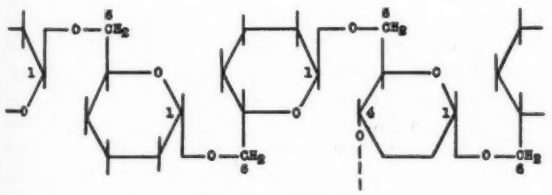


Figure 3. Dextran

Dextran has been approved by the National Research Council for use as a plasma expander. However, important problems remain in the control of molecular size, the tests necessary to produce a suitable product, and the reactions which occur in some patients.

Polyvinylpyrrolidone (PVP or Periston), a synthetic material (Figure 4) prepared from acetylene, formaldehyde, and ammonia, was developed during World War II as the result of extensive screening tests by the Germans for an effective plasma expander. Since the large molecular weight material remains in the body while the small molecular weight material is rapidly excreted, control of molecular size is important. PVP has been approved for stockpiling.

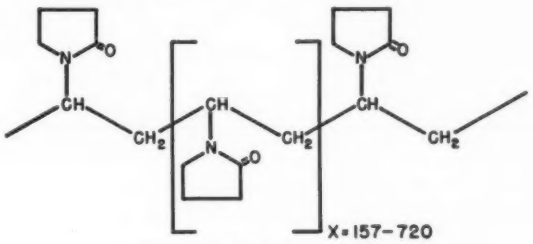


Figure 4. Polyvinylpyrrolidone

Gelatin, a protein material obtained from bone or skin, was one of the first colloidal materials used as a plasma expander. It is a unique protein which does not cause antigenic or foreign protein reactions when injected in humans. Various molecular weight gelatins have been used as plasma expanders. One of these preparations now on the market bears the seal of acceptance of the American Medical Association. This solution is prepared from a high molecular weight (undegraded) gelatin and is a gel at room temperature. Before use, it must be liquified by warming. Solutions of low molecular weight gelatin (degraded by heat) are liquid at room temperature, but are rapidly excreted by the patient and act as plasma expanders for only a short time.

During World War II, a research group under Professors Linus Pauling and Dan H. Campbell, at the California Institute of Technology, succeeded in modifying gelatin so that a 5 per cent solution was liquid at room temperature.[4]

In this process, gelatin is reacted with glyoxal, which couples the gelatin molecules together to form a poly-

[4] Campbell, D. H., J. B. Koepfli, L. Pauling, N. Abrahamsen, W. Dandliker, G. A. Feigen, F. Lanni, and A. Le Rosen, *Texas Repts. Biol. Med.*, 9, 235–80 (1951).

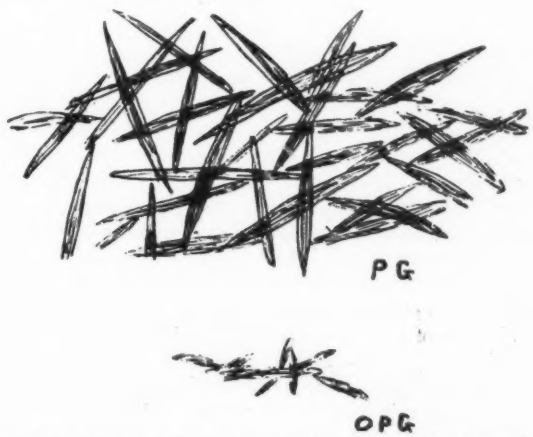


Figure 5. Schematic Illustration of Polygelatin (*PG*) and Oxypolygelatin (*OPG*)

gelatin (*PG* of Figure 5).[5] The polygelatin is then oxidized and degraded with hydrogen peroxide to form a material which they named "oxypolygelatin."

Property	*Gelatin*	*Oxypolygelatin*
Osmotic pressure (cm. of water) at 37°C.	335–405	350–400
Viscosity relative to water at 37°C.	5–7	2.1–2.2
Gel point (°F.)	Above 77°	Below 50°

Modification of gelatin in this manner lowers the viscosity and gel point of the solution as shown in the table. The osmotic pressure, on the other hand is not significantly changed.

Extensive tests in mice, rabbits, dogs, and patients indicate that oxypolygelatin is effective and nontoxic. Elimination of the material from the body, as indicated by urinary excretion, follows much the same pattern as a gelatin solution.

Much work has been done by both chemists and clinicians in developing these various plasma expanders for insurance against the possible disaster of atomic warfare. More harm than good can be done, however, if we fail to keep in mind that these substances are in no way replacements for whole blood.

[5] Campbell, D. H., J. P. Koepfli, and L. Pauling, U. S. Patent No. 2,591,133.

A SIMPLE LECTURE DEMONSTRATION OF PAPER PARTITION CHROMATOGRAPHY

EVELYN S. KRITCHEVSKY and DAVID KRITCHEVSKY[1]
Upsala College, East Orange, New Jersey

This communication describes a demonstration of paper partition chromatography which gives readily interpretable results within the time covered by one lecture period, can be carried out with easily obtainable materials and requires no special solvents, sprays, or developing agents.

The materials required are a large test tube (20 × 2.5 cm.) fitted with a cork, strips of Whatman No. 1 filter paper (17 × 1 cm.), denatured alcohol and a set of food colors which can be purchased in any grocery store. The experiments are carried out using the ascending method of paper chromatography. A few ml. of an alcohol-water solution is introduced into the test tube, the paper strip is suspended in the solvent by stapling it to the under side of the cork stopper and a spot of dye is applied to a point about 3 cm. from the bottom of the paper strip. The strip is suspended so that the spot of dye is about 1 cm. above the level of the solvent. A pin dipped into the dye solution and touched to the paper is the easiest method of applying the spot to the paper. A few staples introduced below the spot keep the paper strip taut. The solvent rises about 5 to 6 cm. during the course of a lecture period, this time being sufficient to give satisfactory results.

The sets of food colors usually available contain vials of red, green, yellow, and blue coloring matter made up of U. S. certified colors in distilled water and alcohol. For our experiments water-alcohol mixtures of 1:1, 6:4, 7:3, 8:2, and 9:1 were used. As the water content of the solvent is increased the colors migrate further along the strip. The three latter dilutions were found to give the best results.

The following results were obtained: The green dye separated into a leading blue and a trailing yellow component; the blue dye into a leading blue zone and a trailing magenta one; and the red dye into a leading pink component and a trailing carmine one. The yellow dye appeared to be homogeneous. The experiment is not quite ideal in that the colors tend to streak, but resolution is rapid and dramatic, especially in the case of the green dye, and the demonstration serves well to illustrate the principles of paper partition chromatography. Using the leading edge of each colored zone, it is possible to illustrate the calculation of Rf values and to show that the blue component of the blue and green dyes and the yellow of the green and yellow dyes are, apparently, identical. The Rf values obtained using water-alcohol 8:2 are given as an example:

Dye	*Component*	*Rf*
Yellow	Yellow	0.32
Green	Yellow	0.32
	Blue	0.87
Blue	Blue	0.88
	Magenta	0.33
Red	Pink	0.53
	Carmine	0.22

The colors available in two different commercial sets were tried, with similar results.

[1] Lederle Laboratories Division, American Cyanamid Company, Pearl River, New York.

Report of the

NEW ENGLAND ASSOCIATION OF CHEMISTRY TEACHERS

OFFICIAL BUSINESS

269th MEETING

The headquarters for the 269th meeting of the NEACT was the Mary C. Wheeler School in Providence, Rhode Island. The school is about two blocks from the Brown University campus and the meeting was opened and the first lecture given in Wilson Hall at Brown.

This first talk was given by Robert T. Beyer, Associate Professor of Physics, Brown University. Dr. Beyer discussed the chemical and physical aspects of ultrasonics. During his talk, he gave a number of demonstrations of ultrasonic principles using a shell of barium titanate as a transducer.

The meeting then adjourned to the Wheeler School.

Wendell T. Caraway, biochemist at the Rhode Island Hospital, was the second speaker. Dr. Caraway talked on the use of the flame spectrophotometer for the study of body chemistry, in particular the balance of sodium ions of the intercellular fluids and potassium ions of the intracellular substances. It was pointed out that the method is responsible for the saving of many lives because its rapid diagnosis allows treatment to be started before it is too late.

After luncheon at the Wheeler School, Leallyn B. Clapp, Associate Professor of Chemistry, Brown University, described the course offered at Brown to first-year students who plan to major in chemistry and who enter with high-school chemistry credit. Dr. Clapp described the course as a study of the covalent bond. Since the best examples of covalent bonding occur in organic compounds, the course is largely organic chemistry. It differs from the usual course in organic, however, in laying more stress on acid-base concepts, and on oxidation-reduction. Students who take this course have no more difficulty in subsequent courses than those who follow a more conventional curriculum.

Business Meeting. President Dorothy W. Gifford conducted the business meeting.

The following members who have joined since the 268th meeting were welcomed into the Association by the Secretary:

Sabion W. Caputo, 26 Lyon Street, Dorchester 22, Massachusetts.

Harry J. Christoffers, Department of Chemistry, University of Massachusetts, Amherst, Massachusetts.

Jean V. Crawford, Chemistry Department, Wellesley College, Wellesley 81, Massachusetts.

Raymond J. Farrell, East Senior High School, Pawtucket, Rhode Island.

Clarke L. Gage, Department of Chemistry, St. Lawrence University, Canton, New York.

Lawrence Goldthwait, St. George's School, Newport, Rhode Island.

Rev. James W. Hackett, O.P., Providence College, Providence 8, Rhode Island.

Enos E. Held, Head of Science, Swampscott High School, Swampscott, Massachusetts.

M. Gertrude McKeon, Connecticut College, New London, Connecticut.

David Palmer Matthews, Bourne High School, Bourne, Massachusetts.

Enice Marth Moore, Mount Holyoke College, South Hadley, Massachusetts.

Gilbert E. Moos, Assistant Professor, St. Lawrence University, Canton, New York.

Edward P. Parry, Department of Chemistry, University of Connecticut, Storrs, Connecticut.

Edward W. Savery, Glen Mills High School, Glen Mills, Pennsylvania.

Sixty-one members have joined since July 1, 1952, bringing the total membership to 545.

The President reported that she had been invited to represent the NEACT at the meeting of the School and College Committee on Advanced Standing, at Wesleyan University in March.

270th MEETING

The 270th meeting of the New England Association of Chemistry Teachers was held on April 11, 1953, at the Teachers College of Connecticut, New Britain, Connecticut. About 50 members and guests were present.

At the morning session, Walter G. Stewart, Chairman of the Western Division, introduced William C. Lee, Dean of the College and a former member of the NEACT, who welcomed the group and gave a brief account of the work and growth of the College, contrasting the present facilities with those available at the time of the last meeting of the Association at Teachers College.

Dr. Hans O. Spauschus of the General Electric Company, in his account of "Instrumental analysis in an industrial laboratory," outlined the types of problems which are brought to the chemist in an industrial laboratory. He gave specific examples and suggested methods of solving them.

This address was followed by a paper on "Chemicals by fermentation," by Jay S. Buckley of Chas. Pfizer

and Co., Inc. After defining fermentation as "any chemical reaction brought about by microorganisms," Dr. Buckley stated that the chemicals to be discussed could be grouped into four categories: (1) those produced by fermentation in competition with synthetic methods; (2) those which may be produced by fermentation, but may be more cheaply produced by synthesis; (3) chemicals whose synthetic production cannot be completed without the aid of microorganisms; (4) chemicals which can be produced only by fermentation. The discussion of these four groups brought into clearer understanding many old friends like citric acid, ethyl alcohol, and acetic acid. It placed in better perspective substances like penicillin and chloromycetin, cortisone and vitamin B_{12}, and it introduced as well some of the newest antibiotics.

During the afternoon session, a color sound film on Krilium was presented. This film gave a good deal of information about the way in which this interesting new product works, and included as well some very beautiful time lapse photography of the blossoming of flowers. At the conclusion of the film Mr. W. P. Sheppard of Monsanto Chemical Company did a number of demonstrations which illustrated in a striking manner the action of Krilium on silty, clay soil.

One of the most interesting features of the day was the inspection of the new Henry Barnard Hall for Arts and Sciences. This very beautiful building (not quite completed at the time of the meeting) has its entire second floor devoted to science. Many teachers present looked with envy at the superb lighting, the adequate and convenient storage spaces, the smooth new table tops, and the generally handsome and efficient classroom equipment. This is a building of which any college could be proud, and it should make the work of teaching far easier and happier and more efficient.

Business Meeting. President Dorothy W. Gifford conducted the meeting. Six new members were welcomed into the Association:

Stanley C. Bunce, Assistant Professor of Organic Chemistry, Rensselaer Polytechnic Institute, Troy, New York.
Roy Glick, Science Teacher, School of Industrial Art Annex, Bronx, New York City.
Earl N. Johnston, Head of Science Department, Williston Academy, Easthampton, Massachusetts.
Walter B. Keighton, Professor of Chemistry, Swarthmore College, Swarthmore, Pennsylvania.
Paul Nickas, Chemistry Teacher, Gloucester High School, Gloucester, Massachusetts.
John W. Sease, Associate Professor of Organic Chemistry, Wesleyan University, Middletown, Connecticut.

SUMMER CONFERENCE

The 15th Summer Conference will be held at Bowdoin College, Brunswick, Maine, August 24 to 29, 1953. President Dorothy Gifford has appointed the following to the conference committee and subcommittees.

Co-Chairmen
John E. Cavelti, Allegheny College, Meadville, Pennsylvania.
Guy F. Burrill, Keene High School, Keene, New Hampshire.

Ex Officio Members
Dorothy W. Gifford, Lincoln School, Providence, Rhode Island.
Dorothy Abel, Brookline High School, Brookline, Massachusetts.
William F. Ehret, New York University, New York, New York.
Marco H. Scheer, Nashua High School, Nashua, New Hampshire.
Constance L. Brown, University of Vermont, Burlington, Vermont.

Representatives of Host Institution, Bowdoin College, Brunswick, Maine.
William C. Root
Samuel E. Kamerling

Registrar and Treasurer
Katherine M. Murphy, Dorchester High School for Girls, Dorchester, Massachusetts.

Secretary
William O. Brooks, Technical High School, Springfield, Massachusetts.

Publicity
Eldin V. Lynn, Massachusetts College of Pharmacy, Boston, Massachusetts.

Speaker Hospitality
Leonard F. Foley, Manchester Central High School, Manchester, New Hampshire.

Social Events
Louise O. C. Swenson, English High School, Lynn, Massachusetts.
Ruth R. Jefferson, Branford High School, Branford, Connecticut.
Angela M. Trovata, Robinson Seminary, Exeter, New Hampshire.
Gerald A. Rose, Brunswick High School, Brunswick, Maine.

Exhibits
Paul F. Davis, Bellows Falls High School, Bellows Falls, Vermont.
Walter W. Holt, Boston University, Boston, Massachusetts.

Motion Pictures
Carl F. Swinnerton, Pomfret School, Pomfret, Connecticut.
Robert D. Eddy, Tufts College, Medford, Massachusetts.

Mailing
Robert F. O'Malley, Boston College, Chestnut Hill, Massachusetts.
Nelson J. Anderson, Suffolk University, Boston, Massachusetts.

Registration Assistants
Alice Howe, Concord High School, Concord, Vermont.
Angelica Courniotes, Western Massachusetts School of Pharmacy, Willimansett, Massachusetts.
Helen A. Pratt, English High School, Lynn, Massachusetts.

Activities
Austin S. Kibbee, Jr., Jay High School, Jay, Maine.

The technical program will include addresses by the following:

William C. Root, Bowdoin College, "Chemistry in Inca archeology."
Harry F. Lewis, Institute of Paper Chemistry, "Sources of cellulose."
William F. Kieffer, College of Wooster, "Chemical problems of nuclear reactions."
J. Frederic Hazel, University of Pennsylvania, "Solid phase reactions."
William Rieman, III, Rutgers University, "Application of ion-exchange to analytical chemistry."
Eugene Ayer, Gulf Research and Development Company, "Future sources of energy."
Edward L. Haenisch, Wabash College, "The uses of various forms of the periodic table."

Otto Eisenschiml, Scientific Compounding Company, "What the employer expects of a graduate chemist" and "A chemical murder case."

Luke Steiner, Oberlin College, and others, discussion of the topic, "Admission to college with advance standing."

Louise Kelly, Goucher College, and others, discussion of the topic, "How to get students to take a more active part in courses in organic chemistry."

Harold E. Coburn, Bulkeley High School, Hartford, Connecticut, illustrated lecture on India.

Members of the Association are anticipating the 15th Summer Conference with great interest. The selection of the 160-year-old Bowdoin College as the site is a happy choice. Bowdoin is a salt-water college, situated close to the rocky Maine coast in a fine vacation area. It has adequate living, dining, and recreational facilities to accommodate the conference and the fine new Parker Cleaveland Laboratory in which to hold the meetings. Moreover, one of the Association's own members has only this year been installed as Bowdoin's president. In addition to the technical program, the committee has arranged an excellent series of social events, including a genuine Maine shore dinner served by a famous Maine caterer. NEACT summer conferences are unique in the large number of wives and families who register in company with their chemistry-teaching husbands, and for the far distances from which many of its guests are attracted. Many western colleagues combine a family vacation trip to New England with attendance at the conference. Anyone interested is invited to inquire of the conference secretary, William O. Brooks, 904 Stony Hill Road, R.F.D. No. 1, Wilbraham, Massachusetts.

To the Editor:

For your amusement will you permit me to answer or rather suggest something to the problem of Dr. Julius Sumner Miller in Letters to the Editor, page 634, December, 1952, in this wise: Will not veterinarian syringes, bulb- or pump-pipets, or the 2-valve syringe used for blood transfusion using Tygon tubing, do?

MARIANO A. HENSON

ANGELES, PAMPANGA
PHILIPPINE ISLANDS

To the Editor:

I believe that the universities should devote specific attention to the graduate student, including the Ph.D. candidate, planning tentatively to teach college chemistry; this training should supplement his present research training. My suggestion is an extension of the graduate teaching assistantship program to stimulate this interest in a college chemistry teaching career.

I would recognize the value of graduate assisting by granting one semester-hour credit for each semester an assistant taught. His grade for that hour would depend upon several pre-announced and extemporaneous observations of his teaching performance by his superior. In addition to the credit recognition I would make it mandatory that the students criticize the assistant's instruction and that these appraisals be compiled each semester into one composite word picture of the assistant's teaching capabilities. An additional word picture would be written by the supervisor and then both read to the assistant and discussed with him, as a means of noting his progress in teaching chemistry. An attempt should be made to rotate assistants in the various fields of chemistry to give them an overall teaching background.

I would initiate a graduate course in chemical education, consisting of ten two-hour periods, for one semester, giving two hours credit. It would be given preferably by guest lecturers from the chemistry department on such topics as: the aims and objectives in college education, special methods of teaching college chemistry, how to give lecture demonstrations, the use of audio-visual aids, standardized tests in college chemistry, etc. Evaluation in the course could consist of a final examination on application of discussed material, and on assigned journal readings in chemical education. This course would provide a chemical education background for those graduate students not on assistantship programs, but who still contemplate college teaching.

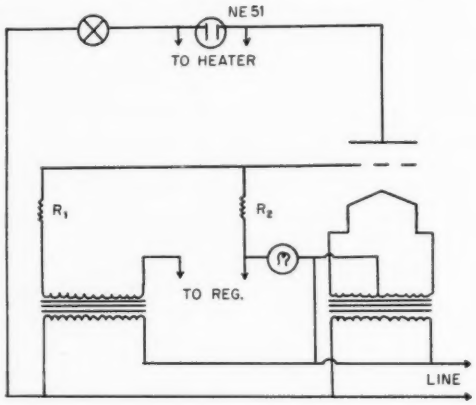
Since the assistantship program is rapidly expanding and is considered an integral part of the program of many graduate students, college credit recognition should be given to it. Also, a course in chemical education should be added to the curriculum to provide a background of sound chemistry teaching principles, for the potential college chemistry teacher.

2ND LT. WALTER KATZ, USAF

ARMY CHEMICAL CENTER
MARYLAND

To the Editor:

Two electronic relays as described by Duncan G. Foster [J. CHEM. EDUC., **29,** 49 (1952)] were recently constructed here. In each device the tube often failed



to "fire" when the regulator circuit was opened. This difficulty is avoided by removing the 100,000-ohm resistor, R_2, from its position in the original circuit and placing it between the thyratron grid and ground, as shown.

As thus modified, this relay appears to serve very well as a temperature control device. We have added an NE 51 neon bulb to indicate whether the heater is on.

H. BRADFORD THOMPSON

MICHIGAN STATE COLLEGE
EAST LANSING, MICHIGAN

To the Editor:

An instructor observing a student pipetting out a solution of sodium arsenite, though previously advised that it was an unsafe procedure, corrected him with words that occurred at the moment: "Buret out, not pipet out poisonous solutions." If "pipet out" has the same meaning as "measure out by a pipet" and if usage can transform a noun into a verb, then "buret out" should be allowed to replace "measure out by a buret."

ARCOT VISWANATHAN

THE MINERVA TUTORIAL COLLEGE
EGMORE, MADRAS, SOUTH INDIA

[EDITOR'S QUERY: Should it?]

● ORGANIC CHEMISTRY

Lucius Junius Desha, **Professor of Chemistry, Washington & Lee University. Second edition. McGraw-Hill Book Co., Inc., New York, 1952. xvi + 595 pp. Illustrated. 16 × 23.5 cm. $6.50.**

WRITING a textbook on organic chemistry is admittedly a very difficult task. In the preface to the second edition Professor Desha says that the book is planned to be the basis for a year course and is "designed particularly for those students whose formal training in the subject will probably end with such a course."

With the wealth of material available since the publication of the first edition in 1936, this solution to the problem of what to include in a beginning textbook is proposed: "to abandon any thought of being comprehensive; to reappraise the entire subject matter, old and new, and select carefully the illustrative material to be used; and, having decided to mention a principle or a product, to treat it in enough detail to give it meaning. The basis of selection and the order of presentation must be, necessarily, somewhat arbitrary. That is part of the price to be paid for a *text*—a book which will give the beginning student a reasonable chance to avoid that sense of utter confusion which Wöhler was experiencing in 1836 when he wrote, 'Organic chemistry gives me the impression of a primeval, tropical forest . . . a monstrous and boundless thicket, with no way of escape, into which one may well dread to enter.'"

This reviewer has the feeling on looking over this text, that the student will still be confused, but that the nature of the forest has been changed. Emphasis is on mechanism of reaction, inductive effect, polarization, etc., at the expense of the behavior of organic compounds. Interrelationships of compounds are understood better by the average student from material presented in charts with references (first edition, page 144) rather than from a list of references to other paragraphs in other chapters (second edition, page 121). Most authors and teachers find it more advantageous to consider the fats and fatty oils along with the fatty acids and other esters rather than as derivatives of polyfunctional alcohols, and to consider aromatic compounds after considerable foundation material of naming and behavior has been built up by the study of simpler compounds.

The topics chosen for discussion are handled well but many teachers will disagree with the choice. Some inaccuracies have been overlooked; for example, on page 75 the statement is made that, "the structure which is common to all compounds classified

as aromatic is an unsaturated six-carbon ring..." Then on page 516 is found, "Aromatic properties are highly developed in pyridine..." On page 45 two aldehydes are named butyric aldehyde and propionic aldehyde and on page 175 the former is called butanal or *n*-butyraldehyde.

The type, binding, and general appearance of the book are good and, for those who prefer to consider the aliphatic and aromatic compounds together, much new material is included on the selected topics.

H. MARJORIE CRAWFORD

VASSAR COLLEGE
POUGHKEEPSIE, NEW YORK

● A SHORT COURSE IN ORGANIC CHEMISTRY

Harold Hart **and** ***Robert D. Schuetz,*** **both of Michigan State College. Houghton Mifflin Co., Boston, 1953. ix + 326 pp. 104 figs. 20 tables. 17 × 23.5 cm. $4.50.**

THIS book is designed for use in a short course for students in agriculture, home economics, pharmacy, and in premedical or predental programs. It is not offered as a text for chemistry majors.

One must not be deceived by a first glance at this book. On quickly leafing through it, one's eyes are caught by photographs of kittens playing with their reflections in a mirror, of a fashion model scintillating as she chats on the telephone, and of a newsstand with giant headlines screaming news of a sensational murder. One also sees "stick-and-ball" models of double and triple bonds surrounded by flashes of lightning. Such glamor suggests that the book is not so much talking chemistry as it is talking about chemistry. This impression is wrong. Closer examination reveals that it is a rigorous textbook within its stated scope.

The authors have, as they should in writing such a book, omitted much material that is commonly taught in a full-year course. For the most part their choice of material is good. A student who had studied from this book, and who then decided to continue the study of organic chemistry, would not be seriously handicapped for lack of background. There is appropriate attention to applications, necessary to maintain the interest of non-majors. The problems at the end of each chapter are excellent, calling for thought on the part of the student and not mere recantation of statements made on earlier pages. There is a profusion of good drawings of molecular models, very useful in teaching stereochemical relationships.

Reaction mechanisms are strongly emphasized. For example, the chapter on olefins discusses, amongst other material, the lack of free rotation about a double bond, the mechanism of addition of bromine to olefins, the theoretical basis of Markownikoff's rule in terms of the inductive effect of the methyl group and its influence on resonance in propylene, the mechanism of 1,2- and 1,4- addition to 1,3-dienes, and the mechanism of free-radical polymerization of olefins. In this reviewer's opinion, this is an overemphasis. So much attention to reaction mechanism tends to confuse the average student who, in the second or third week of the course, is often grappling with problems of nomenclature, has difficulty distinguishing an addition reaction from a substitution or has not yet learned the importance of writing four and only four covalent bonds to each carbon atom.

The style of writing is fast-moving and somewhat abrupt. Its terseness will reduce its value to students of moderate ability.

A few topics are not given adequate attention. Körner's method for distinguishing ortho, meta, and para isomers, an eloquent exercise in scientific method, is not mentioned. Dye chemistry, an aspect of applied organic chemistry of great importance to the students for whom the book is intended, is inadequately treated. The tremendous consequences of stereochemical relationships in life processes are hardly mentioned.

JOSEPH F. BUNNETT

UNIVERSITY OF NORTH CAROLINA
CHAPEL HILL, NORTH CAROLINA

● PROBLEMS IN PHYSICAL CHEMISTRY

Lars Gunnar Sillén, **Professor of Inorganic Chemistry, Royal Institute of Technology, Stockholm, Sweden,** ***Paul W. Lange,*** **Head of Physical Chemistry Division, Swedish Forest Products Research Laboratory, Stockholm, Sweden, and** ***Carl O. Gabrielson,*** **Head of Research Department for Organic Chemistry, Mo and Domsjö AB, Örnsköldsvik, Sweden. Prentice-Hall, Inc., New York, 1952. xi + 370 pp. Illustrated. 15 × 22 cm. $5.50.**

THIS is a book which will be of more than passing interest to teachers of physical chemistry. It is essentially a translation, made by the authors themselves, of the second Swedish edition published in 1951. In bringing out this American edition the authors-translators have taken pains to conform with the symbols and notation in general use on this side of the Atlantic in cases where differences exist. In their effort to do this they have succeeded extremely well.

The arrangement and scope of subject matter are indicated by the 11 chapter headings, as follows: First Law of Thermodynamics, Second and Third Laws of Thermodynamics, Partial Quantities and Activities, Gas Equilibria, Solution Equilibria, Chemical Electromotive Forces, Solubility, Complex, and Redox Equilibria, Acid-base Equilibria, Transport of Electricity by Electrolytes, Rate of Reaction, and Phase Boundaries.

The 900 or more problems in this collection have been gleaned from many sources. For the most part, they are not "'desk' problems in which the quantities asked for would never, in practice, be determined as described," but are based upon actual measurements as reported in the European and American journals in research papers. The *Journal of the American Chemical Society* figures conspicuously as one of the most prolific sources.

The problems are straightforward and clearly stated and are well arranged for purposes of instruction. Their usefulness is enhanced by brief introductions to the several chapters in which the background theory is summarized. These summaries are clear and concise and definitely helpful to the student. Answers are given in the back of the book.

The form and typography of the book are neat. No typographical errors were found, indicating exceptionally careful proofreading.

In the opinion of the reviewer, this is a book which merits careful examination by teachers interested in physical chemistry.

WILLIAM B. MELDRUM

HAVERFORD COLLEGE
HAVERFORD, PENNSYLVANIA

● THE PHILOSOPHY OF SCIENCE

Stephen Toulmin, **Lecturer in the Philosophy of Science, University of Oxford. Hutchinson's University Library, Hutchinson House, London, 1953 (available from Longmans, Green and Co., New York). viii + 176 pp. 12.5 × 19 cm. Text, $1.80; trade, $2.25.**

THIS book is primarily designed for university students in philosophy. Now philosophy has been described as "an obstinate attempt to think clearly." This book uses mostly simple words, and simple mathematics, and simple scientific illustrations, but it splits hairs with considerable subtlety, and is at least as difficult to read as an introductory text in thermodynamics.

Dr. Toulmin has much to say about the principle of rectilinear propagation of light, which is basic to the use of diagrams in considering optical situations, and therefore underlies the whole of geometrical optics. An important hair remains unsplit here, for in the phrase "rectilinear propagation" the two words introduce quite independent ideas. If the velocity of light had been found to be infinite, so that there were no question of anything being "propagated," we could still use rectilinear representations in our diagrams. One wonders, too, why Dr. Toulmin speaks of light as "a substance traveling," when the

word "substance" is applicable neither in its usual meaning in chemistry and other sciences nor in its strict philosophical meaning.

Snell's law (of refraction) is introduced as a Law of Nature, within the context of the rectilinear propagation of light, which is a Principle. Laws of nature require careful statements of the conditions under which they are applicable, such as in this case, "in the absence of temperature variations, and for most isotropic substances."

The "nature statements" of the physical scientist are contrasted with the "habit statements" of the natural historian, as, "ravens are black," "mice are graminivorous." The natural historian, Toulmin says, cannot modify his principles in the light of his discoveries, but the physical scientist can go back and reclassify, or formulate new principles, whenever any discovery makes this desirable. Notice the old-fashioned expression "natural historian." "As soon as an observer suggests, *e. g.*, how the colouring of some sub-species of rat may be explained in terms of its environment, he is promoted from 'natural historian' to the more respectable rank of 'zoologist.'" It is strange that it is the physicist, not the *natural* historian, that makes *nature* statements. It would have been helpful if Toulmin had explained what he understands by "nature."

One of the great problems of philosophy is the problem of universals, or—Can we make a valid induction? Toulmin is of that school of philosophy that shies away from an induction as from a bogey man. Consequently he is at pains to remove physical science as far as possible from "formal logic" (by which he understands only a travesty of the subtle analysis of thought initiated by Aristotle) and leaves the syllogism to the despised natural historian. However, toward the end of the book when he is arguing with all his might against any principle of uniformity of nature, he suddenly introduces a formal syllogism: *major*, "cadmium vapour when excited must emit such-and-such radiation"; *minor*, "this lamp contains cadmium vapour"; *therefore*, etc.

In the opinion of this reviewer, this book, like many others on the same subject, is an application of tenth-rate philosophy to first-rate science. The book should interest all, and will probably please many of those readers of THIS JOURNAL who are genuinely interested in the fundamental philosophy of science.

ANTHONY STANDEN

THE INTERSCIENCE ENCYCLOPEDIA
BROOKLYN, NEW YORK

● CYTOCHEMISTRY

J. F. Danielli, **Professor of Zoology, King's College, London. John Wiley & Sons, Inc., New York, 1953. 139 pp. 8 figs. 10 tables. 12 plates. 14.5 × 22 cm. $4.**

CYTOCHEMISTRY is a young science. As usual in such cases great hopes are attached to new developments which in the future may or may not be fully realized. Many not fully qualified workers are attracted whose uncritical work tends to retard or even discredit otherwise promising trends. In such instances a critical appraisal of the status of a science at a given moment is very desirable and certainly salutory. The present book, the outcome of a series of lectures at the University of Chicago, seems eminently suited to make the overenthusiastic and the uncritical worker ponder. It is written by an expert and is chiefly based on the work he himself and his collaborators have done mainly during the last decade. The author himself emphasizes that it is not intended as a textbook, for which he thinks the time has not yet arrived.

The book is divided into seven chapters. The introductory chapter sets out some general principles and procedures while the next one deals specifically with fixation procedures. It is pointed out that for cytochemical work the freeze-drying method is generally best, and a new inexpensive type of apparatus serving this purpose is described. Chapter 3, dealing with alkaline phosphatase, is quite extensive. It is characterized by the description of a variety of control procedures destined to test for artifacts. The most essential conclusion is the theory that the phosphatase represents the enzymatic center of a contractile protein. Chapter 4 discusses the cytochemistry of aldehydes. The various methods useful for the determination of different types of aldehydes are discussed, together with their rather numerous pitfalls. The following chapter is devoted to proteins and nucleic acids. While in many other phases of cytochemistry more or less modified staining procedures of the classical type are employed, it appears that here the intrinsic absorption spectra, often modified by the use of chromogenic reagents, may become very useful. Chapter 6 deals exclusively with the possibilities of errors encountered in quantitative cytochemistry. The final brief chapter represents an outlook into the future. The author feels that cytochemistry will greatly refine the usual methods of biochemistry and will help to expand the field of comparative biochemistry.

This book should be read by everyone working in the field or contemplating entering it; its truly scholarly and critical approach should be extremely useful in the prevention of errors.

Brief bibliographies are found at the end of the various chapters and the book has a subject index.

THEODOR VON BRAND

NATIONAL INSTITUTES OF HEALTH
BETHESDA, MARYLAND

● ANNUAL REVIEW OF NUCLEAR SCIENCE. VOLUME 2

J. G. Beckerley, **Editor;** ***M. D. Kamen, D. F. Mastick, and L. I. Schiff,*** **Associate Editors. Annual Reviews, Inc., Stanford, California, 1953. ix + 429 pp. 16 × 23 cm. Illustrated. $6.**

IT SEEMS safe to predict that the review article will continue to increase in prominence as the volume of newly published research continues to outstrip our abilities to absorb it in its original form. The possibility, afforded by "Annual Reviews," of maintaining contact with most aspects of a broad field on an almost current basis makes this publication one of the most useful in scientific literature. Those who are familiar with this annually issued volume already know that the articles are prepared by authorities upon invitation by an editorial board.

Unfortunately, it is not possible to comment separately on the 16 articles comprising the 1953 volume. The emphasis in the present volume is decidedly on the side of strictly nuclear phenomena rather than on applications of radioactive substances and chemical effects of radiation. Nevertheless, there are a few articles which deal substantially with such topics.

Four articles in this volume deal with "creation" problems. The specific subjects are concerned with: (1) stellar energy production (E. E. Salpeter); (2) genesis of the elements (R. A. Alpher and R. C. Herman); (3) formation of natural radiocarbon (E. C. Anderson), and (4) origin of cosmic rays (L. Biermann). The nuclear processes giving rise to the several phenomena are of course different either in degree or kind. For those interested in these subjects, these articles alone make the volume well worth having.

The studies of induced nuclear reactions are dealt with in articles on: (1) recent progress in accelerators (E. L. Chu and L. I. Schiff); (2) photonuclear reactions (K. Strauch); (3) high energy fission (R. W. Spence and G. P. Ford), and (4) high energy spallation reactions (D. H. Templeton).

The present status of nuclear forces as deduced from nucleon scattering is covered by G. Breit and R. L. Gluckstern while J. S. Blair and G. F. Chew are the authors of a review of the year's developments in the frenetic field of mesons and heavier subnuclear particles.

Along with meson and kindred particle physics, one of the most active branches of nuclear science currently is that concerned with the identification of nuclear spectroscopic states and decay processes influenced by the nature of the states. An excellent

summary of the present status of theory and experiment on the β-decay process by E. J. Konopinski and L. M. Langer falls into this category as do articles on angular correlations of nuclear radiations by H. Frauenfelder and nuclear moments by B. T. Feld.

The recent improvements in radiation measurement devices has resulted in a marked increase in our knowledge of decay energies. A timely correlation of these decay energies for the β-decay process is presented by C. D. Coryell.

Two articles in this volume deal with chemical effects of nuclear processes. One of these is a review by J. Bigeleisen on isotope (mass) effects on chemical reactions. The other, by G. J. Dienes, is concerned with radiation effects in solids.

There can be no question that an annually issued book such as this fulfills a useful function. The research worker will find periodic critical reviews and bibliographies of all phases of nuclear work. Those with a more casual interest in nuclear phenomena will find it a relatively painless medium for learning the present status of specific topics and the teacher of nuclear physics or chemistry will find such a volume a great time-saver in bringing up to date his lecture notes.

I. PERLMAN

University of California
Berkeley, California

● HEAT TRANSFER PHENOMENA

R. C. L. Bosworth. John Wiley & Sons, Inc., New York, 1952. xii + 211 pp. 41 figs. 13 tables. 14.5 × 22.5 cm. $6.

Dr. Bosworth's book was developed from a general paper presented at the Heat Transfer Conference at Sydney, August, 1948, sponsored by the Australian Branch of the Institute of Physics. As such the monograph is essentially a review of developments up to that time in conduction of heat in gases, radiative transfer, conduction in solids and liquids, forced and natural convection, heat flow with simultaneous phase change, and the use of electrical and other model methods.

The basic concepts of heat transfer are clearly and thoroughly presented. A particularly valuable aspect is the unified viewpoint attained throughout the book by considering heat flow as an example of a transport process. In this way the similarities between diffusion, fluid friction, electric current, and heat flow are continually brought forth.

The book should be a valuable reference to research workers and advanced students in the field. It is not likely to be useful as a direct text in this country because of the lack of problems or illustrations. The emphasis is primarily on the theoretical aspects of heat transfer. No experimental correlation of heat transfer coefficients or description of commercial equipment is found. Practical points such as the magnitude of fouling factors, or scale resistance, are not considered in sufficient detail to be helpful to students interested in industrial heat transfer work.

JOE M. SMITH

Purdue University
Lafayette, Indiana

● THE MICROBIOLOGICAL ASSAY OF THE VITAMIN-B COMPLEX AND AMINO ACIDS

E. C. Barton-Wright. Pitman Publishing Corp., New York, 1952. x + 179 pp. 25 figs. 30 tables. 14 × 22 cm. $4.

Analytical biochemistry receives a useful tool in this succinct yet comprehensive manual of assay techniques in microbiological chemistry. Beginning with a brief history of microbial requirements for vitamins and growth-factor substances, the author outlines the principles of microbiological assay and presents statistical procedures for computing and testing the validity of such determinations.

The major portion of the book is devoted to specific techniques for determination of the B vitamins and of amino acids. In each instance, consideration is given to the test organism of choice, the basal medium, and the assay procedure (which includes preparation of the inoculum, preparation of samples for assay, and methods of calculation). Vitamins determined include the following: riboflavin, nicotinic acid and nicotinamide, pantothenic acid, biotin, aneurin, folic acid, vitamin B_6-complex, inositol, choline, and *p*-aminobenzoic acid. The section on amino acids is devoted to assay procedures for the following compounds: leucine, isoleucine, and valine; lysine; phenylalanine; histidine; serine, proline and glycine; arginine and threonine; aspartic acid; α-alanine; methionine, cystine, and tyrosine; tryptophan; and glutamic acid.

Author and subject indexes are attached. Usefulness of the manual is enhanced by inclusion of appended information on culture maintenance and preparation of stock solutions. The book is well documented, but literature citations do not include material more recent than 1950.

WILLIAM D. ROSENFELD

California Research Corporation
La Habra, California

● THE ESSAYS OF JEAN REY

A facsimile reprint of the original edition of 1630 with an introduction by *Douglas McKie,* Reader in the History of Science in the University of London. Longmans, Green and Co., New York, 1952. lxxxiii + 144 pp. Illustrated. 13 × 19 cm. $5.

"The Essays of Jean Rey, Doctor of Medicine," on the increase in weight of tin and lead on calcination is known to most chemists as the first publication in which the conversion of the metals into the oxides is attributed to the action of the air. An excellent English translation of the preface by Rey and the 28 short essays and conclusion has been available since 1904 as Alembic Reprint No. 11. But Alembic Reprint No. 11, valuable as it is for students interested in the history of chemistry and in seventeenth-century thought, does not include the dedication to the Prince of Sedan, An Ode on the Essays by Bereau, some verses on the work by Deschamps, and the letter of Pierre Brun, Master-Apothecary in Bergerac, which, according to Rey, was the occasion for the hours he spent on the experiments and those he spent on building up by reason his explanation of the results. The substance of the Essays can be had from Alembic Reprint No. 11, but the charm of the writing as it came from the pen of this early seventeenth-century physician, conversant with many aspects of the life and science of his time is lacking. Copies of the original are very rare, there being only seven extant, one of which is in the Smithsonian Institution, Washington.

To add to the value of this facsimile edition, Dr. McKie has written a fine introduction which includes a life of Jean Rey and short summaries of the contents of the essays; and has added an appendix of six important letters: two of Mersenne to Rey, two of Rey to Mersenne, and two of Brun to Mersenne. In the letter, dated January 1, 1632, to Père Marin Mersenne, "the Minorite father, already entertaining correspondence with the savants of Europe and shortly to embark on those weekly conférences where the latest news from the scientific front was regularly considered and discussed," Rey describes the thermometer he has invented (the ancestor of our present-day thermometer) to use in his practice of medicine. Of it he writes: "it is merely a small round phial having a long and slender neck. For my use I put it in the sun and occasionally in the hand of one with fever, having filled it with water except for the neck, the heat dilating the water makes it rise: the more or less indicating to me great or little heat . . . "

In the first 15 essays, Rey, according to his own statement, lays the foundation in reason for the explanation that is given in the most important essay, number 16: "this increase in weight comes from the air, which in the vessel has been rendered denser, heavier, and in some measure adhesive by the vehement and

long-continued heat of the furnace: which air mixes with the calx (frequent agitation aiding) and becomes attached to its most minute particles: not otherwise than water makes heavier sand which you throw into it and agitate, by moistening it and adhering to the smallest of its grains." There is no suggestion anywhere in these essays, nor in any of the writing in the seventeenth century, on the calcination of metals as a combination of air with the metal. Rey emphasizes that the air has weight; heat from a furnace or from a burning glass can render air denser and thus add weight to the calx.

Dr. McKie in the introduction gives three copies of Jean Rey's rather unusual signature. The one plate is a copy of his inscription on matriculating in the University of Montpellier.

The book is an attractive small volume bound in a wine-colored cover of cloth and paper. The printing is excellent.

CLARA DE MILT

Newcomb College, Tulane University
New Orleans, Louisiana

● WOOD CHEMISTRY. VOLUMES I AND II

Edited by *Louis E. Wise*, The Institute of Paper Chemistry, Appleton, Wisconsin, and *Edwin C. Jahn*, State University of New York, College of Forestry, Syracuse. Second edition. Reinhold Publishing Corp., New York, 1952. Vol. I: ixl + 688 pp. Vol. II: ix + 653 (691–1343) pp. Illustrated. 15.5 × 23.5 cm. $15 per volume.

This treatise ranges from the growth, anatomy, and physical properties of wood, the components and chemistry of the cell wall, and the extraneous components of wood in the first volume through the surface properties of wood, its chemical utilization, its biological decomposition, and its chemical analysis in Volume II. In many ways it is an expansion of the familiar first edition which has been installed as the principal reference book on the subject these last eight years on the bookshelves of everyone interested in wood.

The contributors, over a score of them, are a list of the experts on this subject, so important from its scientific aspects and so important from its industrial aspects. The science of wood chemistry, its technology and applications, covers, by these interrelations with almost every other field of human knowledge, much that is important to our life and civilization. Not everyone of these interrelations can be given in the detail which it might deserve; but the selection of the editors and the contributors of these aspects to be included and the compression of them into a space of only a little over 1,300 pages is remarkable and agrees generally with this reviewer's idea of relative importance.

Many of the additions and modifications of the present volume have been due to the use throughout the world of the earlier presentations and the suggestions which have been received for expansion. Furthermore, in these years since the first edition, there have been entirely new fields which required attention because of these developments.

The printing is excellent; the paper itself, as should be expected in such a book of wood chemistry, is excellent; there are somewhat less than the expected number of illustrations owing to the fact that this is primarily a book on science rather than on the mechanics of wood technology; and there is an excellent subject index of 32 pages, so necessary where so many aspects of a given subject must be considered. The dust jacket blurb—never intended to put the worst foot forward—in this case may be taken at face value: "All workers and students in such fields as pulp and paper, rayon, cellulose films, lacquers and plastics, wood technology, botany and agricultural chemistry, will gain from this valuable monograph a solid foundation of basic wood chemistry and the penetrating insight into the application of this knowledge to modern industrial process."

The first book of this series was the "Chemistry of Wood" by Hawley and Wise published in 1926 and containing 311 text pages; the second member of the series and the first with the present name, "Wood Chemistry," was by Wise alone in 1944 and contained 860 text pages. The present, 1952, volume contains 1343 pages after an interim of eight years and now carries a second distinguished name, that of Dr. Jahn, on the masthead. One might try to extrapolate from this series of 311, 860, and 1343 pages and find that future editions will carry many more pages on wood chemistry. Certainly the rate is accelerating and certainly most of those which will be written in the future will be by men who will be using as their background the material contained in the present volumes.

DONALD F. OTHMER

Polytechnic Institute
Brooklyn, New York

● ADHESIVES FOR WOOD

***R. A. G. Knight*, Officer-in-Charge, Composite Wood Section, Forest Products Research Laboratory, Princes Resborough. Chemical Publishing Co., Inc., New York, 1952. xi + 242 pp. Illustrated. 14.5 × 22 cm. $5.**

This book is an American reprint of an earlier English book written principally from the standpoint of an engineer desirous of joining components mechanically. There has been an attempt made to cover practices in other countries besides England, particularly the various parts of the British Commonwealth; but some of the more recent American practices and developments are not included. Although it deals specifically with fastening woody components, it can be considered as a ready reference for most of the known adhesive types.

Two general phases of the problem have been considered: first the choice of a suitable material for specified conditions of service and secondly, or inversely, the answer to the question as to whether this is the best possible performance in service of the chosen joint medium. After a brief introduction of the reader to adhesives, their theory and classification as natural and synthetic materials, the author discusses the various factors in gluing techniques and such controlling factors as pressure, moisture, cleanliness of surface, pH, temperature, permeability, etc. There is another section on the conditions under which adhesives are used including the moisture content relations of wood, behavior of wood in sheltered environments, and behavior in exposed environments such as boats, aircraft, etc. A final major section of testing of adhesives is followed by a classification of glues and discussion of bonding of wood to other materials. There is a glossary of special terms and a comprehensive index.

This will be a popular handbook for all interested in industrial applications in this field.

LOUIS G. RICCIARDI

Polytechnic Institute
Brooklyn, New York

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By W. THEILHEIMER.
Volume VII (in English): 464 pages $14.90

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(This is the fifth volume in the series, *The Chemistry of Heterocyclic Compounds*, A. Weissberger, Consulting Editor.)

MOLECULAR THEORY OF FLUIDS

By H. S. GREEN, *Institute of Mathematical Physics, University of Adelaide, S. Australia.* 1952. 6 x 9. 288 pages, 12 illus. $5.75
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MECHANICAL PROPERTIES OF WOOD AND PAPER

Edited by R. MEREDITH, *College of Technology, Manchester, England.* 1953. 6 x 9. 300 pages, 88 illus. $7.25
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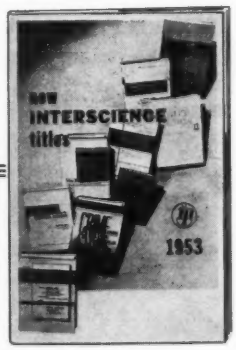

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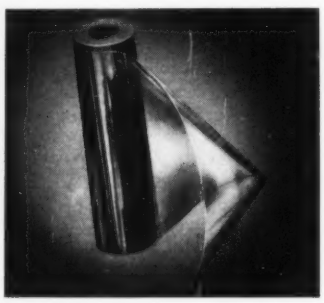

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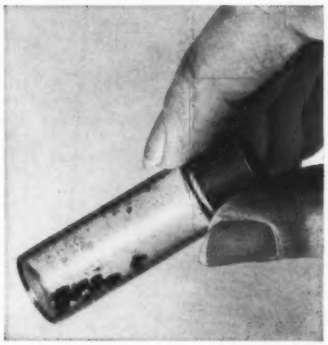

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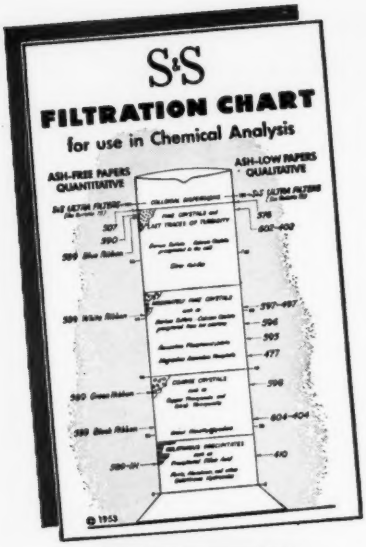

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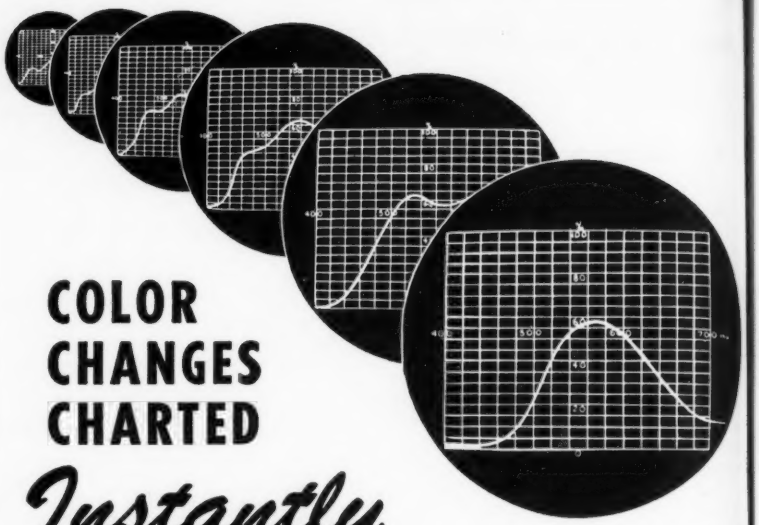

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Electronic Structure Chart

We have received word that the chart of the Electronic Structure of the Atoms, which appeared on pages 576–7 of our December, 1944, issue, may be had from the author, Dr. Don DeVault, College of the Pacific, Stockton, California.

Sublimation Apparatus

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The World List

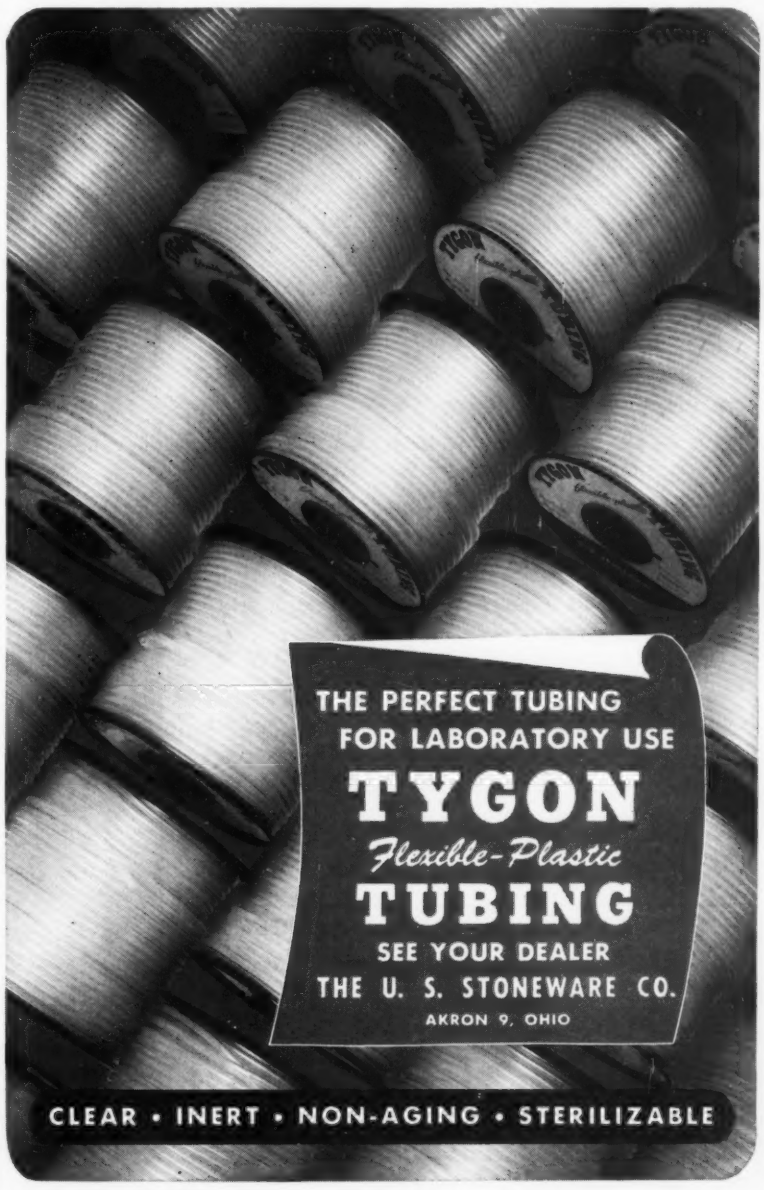
Academic Press, Inc., has released in this country a revised and completely reset third edition of "The World List of Scientific Periodicals."

More than 30 years ago the British Museum started to compile a list of all scientific periodicals together with internationally agreed abbreviations of titles. The first two editions of "The World List" served to establish the volume as a standard work of reference and as an indispensable research tool for editors and librarians in all fields of science.

The new edition lists more than 50,000 volumes, representing an increase of nearly 40 per cent over the second edition, and includes titles of new journals published between 1930–50. The cloth bound volume is available at $37.

Indicating Papers

The Fisher Scientific Co., Pittsburgh, Pennsylvania, has produced a kit of six "short-range" indicating papers which divide the pH scale of 0 to 14 into distinctive colors for each half unit and permit estimations to one-fourth unit. It is recom-



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Description of New Model 14

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Article by Van Z. Williams

USE OF KBr FOR INFRARED SOLIDS ANALYSIS
Description of Revolutionary New Technique

Among the subjects from the Winter '53 issue are:

CONTINUOUS PROCESS STREAM INFRARED ANALYZERS
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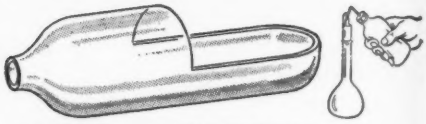

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Arapahoe Chemicals, Inc., Boulder, Colorado, has announced the availability of 2,5-diphenyloxazole, in laboratory and pilot quantities, for use in liquid scintillation counters. It has the advantage of far greater solubility at low temperatures than other scintillants formerly used.

Recovery of Silver

From one of our correspondents in the Philippine Islands, Moises S. Soriaga, comes the following suggestion of a method for the recovery of silver from silver precipitates.

Treat the washed silver precipitates with a fairly concentrated solution of NaOH and cane sugar. Heat gently with constant stirring. The precipitate peptizes, a silver mirror forms, and, finally, metallic silver (with some occluded silver salts) precipitates and settles at the bottom of the container.

Decant the mother liquor and wash with acidified distilled water. Dissolve the precipitate by heating with the least amount of concentrated nitric acid. If solid $AgNO_3$ is desired, filter through an asbestos filter, receive in a shallow dish, and store until dried in a dark closet. If a solution is desired, heat to decompose the excess acid, dilute to the desired concentration (1 ml. of concentrated nitric acid used yields about 10 millimoles of silver nitrate) and filter. The precipitates may be combined with the next batch of silver residues to be treated.

Silver nitrate recovered in this manner is of reagent grade for undergraduate work. Under certain circumstances this method may have some advantages over the usual fusion of silver chloride with sodium carbonate.

New Literature

● The current issue of *Laboratory Spotlight* describes and illustrates a number of new products for the laboratory. Copies of this attractive booklet are available from Harshaw Scientific, Division of the Harshaw Chemical Co., 1945 E. 97th St., Cleveland 6, Ohio.

● A new folder titled *"Norelco X-ray Diffraction School"* gives the history, the objectives, the subjects, the schedule, and the names of lecturers for the week-long free X-ray Diffraction School sponsored semiannually by the Research & Control Instruments Div., North American Philips Co., Inc., 750 S. Fulton Ave., Mount Vernon, New York.

The folder contains a franked mailing card for the convenience of scientists who wish to register tentatively for similar educational forums which Philips will hold in the fall of 1953 and spring of 1954.

● An electronic repeat cycle timer suitable for laboratory testing such as heat cycling and refluxing, is described in a leaflet available from G. C. Wilson & Co., 2 N. Passaic Ave., Chatham, New Jersey.

● Bulletin No. 20, titled " 'Elphco' electronic Relay, Operating Procedure," contains technical information as well as the complete operating procedure for the new "Elphco" Electronic Relay. Requests for copies should be addressed to Electro-Physics Co., 287 Broadway, New York 7, New York.

● A new catalog of isotopes, both radioactive and stable, produced by Oak Ridge National Laboratory, Oak Ridge, Tennessee, is now available, according to an announcement by the Laboratory. The catalog replaces a previous edition published by the Atomic Energy Commission in March, 1951.

More than 100 radioactive and 175 stable isotopes are listed in the new catalog, together with prices and descriptive material. Prices range from as low as $5.00 per curie for radioactive cobalt (in large quantities) to $5.00 a microcurie for radioactive chlorine.

The catalog—at $1.00 per copy—or information about ORNL-produced radioisotopes, may be obtained by writing to Carbide and Carbon Chemicals Company, Oak Ridge National Laboratory, Radioisotope Sales Department, Post Office Box P, Oak Ridge, Tennessee. Correspondence regarding stable isotopes should be directed to Carbide and Carbon Chemicals Company, Oak Ridge National Laboratory, Stable Isotope Research and Production Division, Post Office Box P, Oak Ridge, Tennessee.

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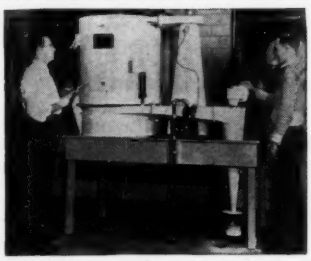

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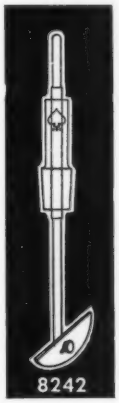

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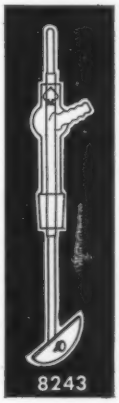

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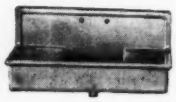

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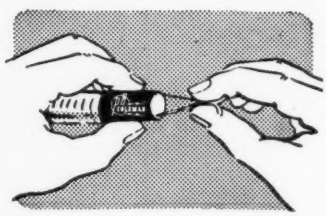

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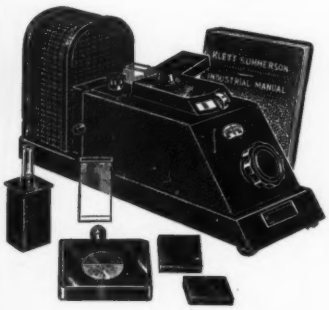

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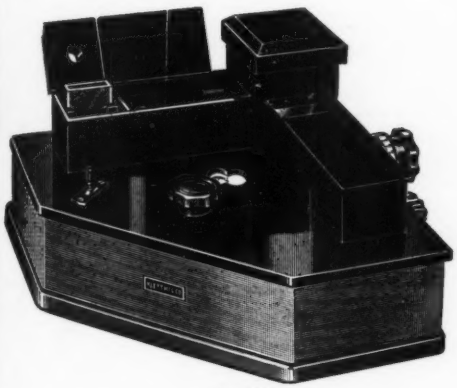

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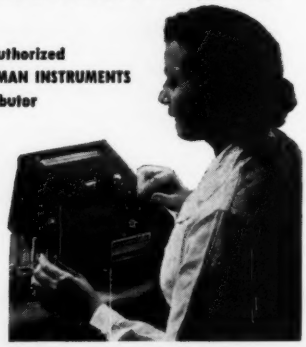

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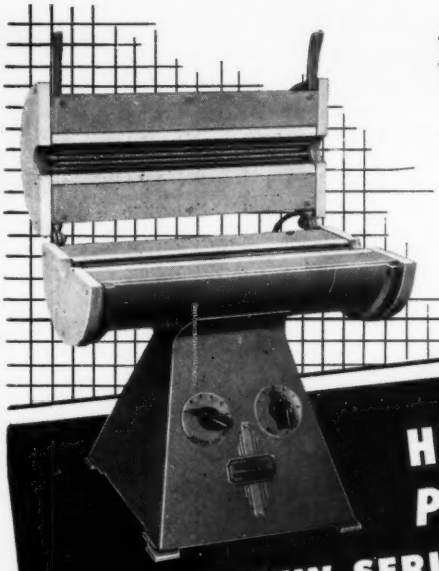

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